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INDEX TO VOLUME XXXVIII.

INDEX OF NAMES.

ABDERHALDEN, E., Electrically Heated Apparatus for Measuring Rotatory Power at Constant Temperature, 300
————, *and* Fuchs, D., Accuracy of the Estimation of *l*-Tyrosine in Proteins, 219
Abel, E., Copper in Distilled Water, 474
Abelmann, A., *and* Rosenthaler, L., Mercuric Oxide as a Standard for Volumetric Analyses, 439
Acland, L. H. D., *and* Rideal, S., Examination of the Oils from *Manihot Ceara* and *Funtumia Elastica*, and a Comparison of their Properties with those of Linseed and *Hevea* Oils, 259
Adams, L. H., *and* Johnston, J., Improved Pyknometer for Density of Solids, 130
———, M., *and* Holmes, A., Pine-Nut Oil, 275
Ageton, C. N., *and* Gile, P. L., Significance of the Lime-Magnesia Ratio in Soil Analyses, 126
Agraz, J. S., Apparatus for Gas Analysis, 395
Agulhon, H., Detection of Volatile Fatty Acids, 274
Aitchison, L., *and* Ibbotson, F., Determination of Copper in White Metal Alloys, 222
Allemann, O., Estimation of Fat in Cheese, 417
Allen, I. C., Preparation of Specifications for Petroleum Products, 335
————, *and* Robertson, J. W., Determination of Sulphur in Fuels, Especially Petroleum Products, 38
————, *and* Jacobs, W. A., Estimation of Water in Petroleum and its Products, 165
Altmann, P., Detection of Nitrous Acid in the Presence of Ferric Salts, 289
Amon, F. O., Lee, R. E., *and* Uhlinger, R. H., Qualitative Analysis of the Zinc Group, 349
Anderson, J. F., *and* McClintic, T. B., Method of Standardising Disinfectants with or without Organic Matter, 422
————, R. P., Portable Pettersson-Palmqvist Apparatus, 178
Angelico, F., *and* Catalano, G., Occurrence of Formaldehyde in the Juices of Plants, 152
Apitsch, H., Estimation of Sulphur in Organic Substances, 515
Archbutt, L., A Simple Test for Differentiating between Cocoa Butter and "Green Butter," 203
————, Examination of the Oils from *Manihot Ceara* and *Funtumia Elastica* and a Comparison of their Properties with those of Linseed and *Hevea* Oils, 263
————, The Bacterial Testing of Disinfectants: A Practical Criticism, 198
————, The Detection and Estimation of Nickel by Means of α-Benzildioxime, 320
————, The Detection of Adulteration in Linseed Oil, 7
————, The Preparation of Rubber for Analysis, 550

Archbutt, L., The Proportionate Determination of Cocoanut and Palm-Kernel Oil in Mixtures, 258

Armani, G., *and* Barboni, J., Examination of Honey, 20

———, *and* Rodano, G. A., Estimation of Paraffin Wax and Ceresin in their Mixtures, 37

Arnold, V., Nitroprusside Reaction of Urine, 154

Arny, H. V., International Standards for Coloured Fluids, and a Suggested Plan for Such Standardisation, 394

Aron, F., New Indicator, 427

——, H., Extraction Apparatus for Solids and Liquids, 351

Arpin, M., Farines, Fécules et Amidons, *Review*, 545

Artmann, P., *and* Preisinger, J., Estimation of Phosphorus in Iron and Steel, 291

Aschan, O., Formic Acid as a Solvent, 513

Astruc, A., Assay and Estimation of Aspirin, 414

Atack, F. W., A New Method for the Volumetric Estimation of Chromium, Vanadium, and Iron, in Presence of One Another, 99

———, Determination of Stannous Chloride and of Chlorates by Means of Methylene Blue, 126

———, The Detection and Estimation of Nickel by Means of α-Benzildioxime, 316

Auger, V., New Method for Volumetric Estimation of Uranium, 230

Auguet, A., Estimation of Essential Oils in Absinthe and Similar Liqueurs, and in Alcoholic Solutions of Essential Oils, 418

Auld, S. J. M., Cyanogenesis under Digestive Conditions, 568

———, *and* Mosscrop, J. D., Measurement of Tryptic Protein Hydrolysis by Determination of the Tyrosine Liberated, 153

Autenreith, W., *and* Funk, A., Colorimetric Methods for Water Analysis by the Use of Autenreith-Koenigsberger's Colorimeter, 179

Bailey, E. H. S., *and* Peterson, G. O., Hickory Nuts and Hickory Nut Oil, 498

Bainbridge, J. S., *and* Davies, S. H., Essential Oil of Cocoa, 20

Baker, H. A., Iodine Titration Method for the Estimation of Tin, Especially in Connection with the Estimation of Tin in Canned Foods, 23

Bakker, C., Estimation of Petroleum Spirit in Turpentine, 464

Bang, I., Method of Estimating Sugar, 218

Baragiola, W. I., *and* Boller, W., So-Called Non-Alcoholic Wines of Commerce, 565

Barboni, I., Analysis of Commercial Calcium Citrate, 65

———, *and* Villavecchia, V., Estimation of Boric Acid in Charcuterie, 55

Barboni, J., *and* Armani, G., Examination of Honey, 20

Bardach, B., Detection of Blood, 568

———, *and* Silberstein, S., Estimation of Glycerol in Sweet Raisin Wines, 24

Barnebey, O. L., *and* Wilson, S. R., Application of Diphenylcarbazide ("Diphenylcarbohydrazide") as Indicator in Titration of Iron with Dichromate, 167

Barth, R., Detection of Nitric Acid (Nitrates) in Milk, 563

Barthélemy, H., Analysis and Examination of Non-Inflammable Cellulose Esters. Acetylcelluloses, 577

Bartlett, E. P., *and* Forbes, G. S., Electrometric Titration of Dichromate Ion with Ferrous Iron, 585

Bass, R., *and* Wiechowski, W., Estimation of Purine Bases in Blood, 107

Baudisch, O., *and* Klinger, G., New Gasometric Method for the Estimation of Nitric Oxide: I., 45

Beadle, C., The Preparation of Rubber for Analysis, 553

———, *and* Stevens, H. P., A Simple All-Glass Extraction Apparatus, 143

Beck, C., Estimation of Copper as Sulphide, 582

Bolton, E. R., The Detection and Estimation of Nickel by Means of α-Benzildioxime, 320

———, The Detection of Adulteration in Linseed Oil, 7

———, The Determination of Tannin in Tea, 316

———, The Proportionate Determination of Cocoanut and Palm-Kernel Oil in Mixtures, 258

———, *and* Revis, C., A Simple Test for Differentiating between Cocoa Butter and "Green Butter," 201

Bömer, A., Mixed Glycerides of Palmitic and Stearic Acids Present in Lard, 204

———, *and* Limprich, R., Nomenclature of Mixed Glycerides and the Synthetic Preparation of α-Distearin and β-Palmitodistearin, 214

———, Polenske "Difference Value" for the Detection of Certain Animal Fats and its Theoretical Basis, 204

Bonnes, L., Detection of Formic and Acetic Acids, 275

Bosshard, E., *and* Grob, W., Analysis of Hydrosulphites, 284

———, New Process for Titration of Sulphurous Acid and of Sulphites in Presence of Thiosulphates, 297

Boswell, M. C., Direct Estimation of Oxygen in Organic Compounds, 216

Bosworth, A. W., *and* Folin, O., Application of Folin's Method for the Determination of Ammonia to Fertilisers, 381

Böttcher, W., *and* Schürmann, E., Estimation of Arsenic in Pyrites, 117

Boughton, E. W., Effect of Certain Pigments on Linseed Oil. Manganese Content of Raw Linseed Oil, 215

Bourdillon, R., Preparation of Conductivity Water, 346

Bourion, F., *and* Deshayes, A., Quantitative Separation of Chromium and Aluminium: Analysis of Chromite, 473

———, Quantitative Separation of Iron and Chromium, 387

Brach, H., Modification of the Liebig Combustion Method, 301

Bradbury, W. A., *and* Owen, F., Estimation of the Carbonates and Hydroxides of Potassium and Sodium when together in Solution, 166

Breed, R. S., Influence of Centrifugal Action on the Distribution of Cell Elements in Milk, 108

Brisleo, F. J., An Introduction to the Study of Fuel, *Review*, 488

Brock, F. P., Redman, L. V., *and* Weith, A. J., Drying Rates of Raw Paint Oils, 468

———, Effect of Temperature, Acid Concentration, and Time in the Bromination of Phenol for Quantitative Determinations, 335

Browne, C. A., A Handbook of Sugar Analysis: A Practical and Descriptive Treatise for Use in Research, Technical, and Control Laboratories, *Review*, 235

———, Combination Specific Gravity Bottle and Dilatometer, 485

Browning, P. E., Introduction to the Rarer Elements, *Review*, 240

Brunck, O., Estimation of Small Quantities of Carbon Monoxide, 75

Bruno, A., Beurres et Graisses Animales (Manuels Pratiques d'Analyses Chimiques), *Review*, 402

Buchtala, H., Method for the Estimation of Mercury in Urine and Animal Tissues, 153

Budai, K., Quantitative Estimation of Trimethylamine in Presence of Ammonia, 436

Bulir, J., Saffron Containing Formaldehyde, 420

Bunge, C., Action of Baryta Water on Certain Sorts of Glass, 118

Burdick, W. L., *and* Gooch, F. A., Electro-Analysis with Light Platinum Electrodes, 43

Burgess, G. K., Micropyrometer, 130

Beck, C., Iodimetric Estimation of Iron, 586

Beckers, W., Comparison of the Farup and Schumacher-Jung Methods Estimation of Mercury in Urine, 111

Beer, R., *and* Meyer, H., Composition of Arachis Oil, 514

Behre, A., *and* Frerichs, K., Composition of Vermouth Wines, 267

Bekk, J., Cerium Dioxide as Contact Substance in Analysis by Combustion, 519

Belasio, R., Detection of Antimony in Alloys, 42

Bell, J. M., *and* Cowell, C. F., Methods for the Preparation of Neutral Solutions Ammonium Citrate, 116

Bellet, A., Estimation of Lactic Acid, 379

Bellucci, J., *and* Grassi, L., Use of "Cupferron" in the Separation of Titanium fro Aluminium, 390

Beolio, L., *and* Lowenstein, A., Experiments on the "Cold Test" of Oil, 34

Berberich, F. M., Burr, A., *and* Berg, A., Composition of Colostrum and of Colostru Fat, 107

Berg, A., Burr, A., *and* Berberich, F. M., Composition of Colostrum and of Colostru Fat, 107

Berrár, M., Studies on the Chemistry of Gelatin and its Estimation, 67

Berry, E. H., *and* Winton, A. L., Chemical Composition of authentic Vanilla Extracts, together with Analytical Methods and Limits of Constants, 206

Bertainchand, E., Estimation of Glycerol in Wines, 368

Bertram, S. H., New Method of Estimating Glycerol, 275

Bertrand, G., *and* Weisweiller, G., Volatile Constituents of Roasted Coffee, 417

Besson, A. A., Estimation of Rosin in Soap, 280

————, Estimation of Thiosulphate in Presence of Sulphites, 481

Bevan, —, The Preparation of Rubber for Analysis, 554

Beyersdorfer, P., Estimation of Sugars by the Kjeldahl-Bertrand Method, 218

Bhattacharyya, H., Rapid Method for Determination of Phosphorus in Steel, 442

Bielecki, J., *and* Henri, V., Quantitative Study of the Absorption of Ultra-Violet Rays by the Fatty Acids and their Isomeric Esters, 113

Bierry, H., *and* Gruzewska, Z., Estimation of Glycogen in Liver, 63

Billy, M., Determination of the Specific Gravity of Mineral Powders, 296

Biltz, W., *and* Marcus, E., Titration of Phosphoric and Boric Acids, 124

Binder, K., *and* Weinland, R. E., New Delicate Reaction for Elementary Oxygen, 172

Birchby, J. A., *and* Merriam, E. S., Methods for the Estimation of Natural Gas for the Production of Gasoline, 577

Blacher, C., Grünberg, P., *and* Kissa, M., Use of Potassium Palmitate in Water Analysis, 127

Black, J. W., Note on the Estimation of Chlorine in Rag Flock, 409

Blomquist, A., Estimation of Mercury in the Air of Rooms, etc., where the Metal is Used, 477

Blum, F., *and* Grützner, R., Estimation of Iodine in Organic Substances, 427

Blumenthal, P. L., Preparation of Selenic Acid and Sodium Selenate as Reagents for the Estimation of Bromine in Halogen Salts, 295

————, *and* Gooch, F. A., Application of Selenic Acid to the Estimation of Bromide in Presence of Chloride, 227

————, Iodic Acid Method of Estimating Bromine in Halogen Salts, 166

Bodmer, R., The Detection of Adulteration in Linseed Oil, 7

Bogdóndy, S. v., Determination of Chlorides and Bromides in Organic Liquids, 211

————, Determination of Pepsin Activity, 210

Boiry, F., *and* Thomas, V., Baobab Oil, 498

Boller, W., *and* Baragiola, W. I., So-Called Non-Alcoholic Wines of Commerce, 565

Bolton, E. R., A Simple All-Glass Extraction Apparatus, 144

Burgess, W. T., Constant Temperature Apparatus, 311
Burgstaller, A., *and* Rothmund, V., Estimation of Ozone and Hydrogen Peroxide, 344
Burmeister, H., *and* Hoepfner, W., Examination of Tung Oil, 116
Burnett, H. R., *and* Revis, C., The Proportionate Determination of Cocoanut and Palm-Kernel Oil in Mixtures, 255
Burr, A., Berberich, F. M., *and* Berg, A., Composition of Colostrum and of Colostrum Fat, 107
Burrell, G. A., *and* Seibert, F. M., Apparatus for Gas Analysis Laboratories at Coal Mines, 482
——————————————, Apparatus for the Exact Analysis of Flue Gas, 351
Büttner, E., Estimation of Water-Soluble Phosphates in Ammonium Superphosphates, 392

Cain, J. R., *and* Tucker, F. H., Determination of Phosphorus in Steels Containing Vanadium, 478
Calvet, L., Alcool Methylique, Vinaigres, *Review*, 352
Camilla, S., *and* Patusi, C., Detection and Estimation of Xanthin Bases in Cocoa, Tea, and Coffee, and their Preparations, 26
——————, *and* Pertusi, C., Analysis of Ferro-Silicon, 76
——————————————, Detection and Estimation of Xanthine Bases in Cocoa, Tea, Coffee, and their Preparations, 60
Campbell, C., *and* Parker, A., Preparation and Analysis of Methane, 430
——————, F. H., Determination of Moisture in Organic Substances, 177
——————————, Separation of Iron and Manganese, 121
Campion, A., *and* Ferguson, J. M., Preparing Sections of Fractures of Steel for Microscopic Examination, 528
Cappelli, A., *and* Silvestri, G., Detection of Nickel in Plated Articles, 45
Carpiaux, E., Source of Error in the Kjeldahl-Gunning Method, 467
Catalano, G., *and* Angelico, F., Occurrence of Formaldehyde in the Juices of Plants, 152
Ceccherelli, A., *and* Fortini, V., Testing Parchment Papers, 161
Černý, F., Melnik (Bohemian) Wine, 326
Chancel, F., Small Apparatus for Electrochemical Analysis, 179
Chaney, W. K., Electrolytic Deposition of Antimony, 581
Chapman, A. C., Action of Tartaric Acid on Tin in the Presence of Oxygen, 472
——————————, An Electrochemical Indicator for Oxidising Agents, 362
——————————, Examination of the Oils from *Manihot Ceara* and *Funtumia Elastica* and a Comparison of their Properties with those of Linseed and *Hevea* Oils, 263
——————————, Nitrogenous Constituents of Hops, 459
——————————, The Bacterial Testing of Disinfectants : A Practical Criticism, 198
——————————, The Determination of Tannin in Tea, 315
——————————, The Preparation of Rubber for Analysis, 554
Chavan, P., *and* Duserre, C., Estimation of Phosphorus in Manures and Feeding-Stuffs, 563
Chavanne, G., Application of Young's Method for the Preparation of Absolute Alcohol, 424
Chelle, L., *and* Denigès, G., New Reagent for Bromine and Chlorine, 119
Chenu, G., *and* Leduc, E., Matériaux de Gros-Ouvre, *Review*, 181
Chercheffsky, N., Analysis of Amyl Acetates and Collodions, 365
Chick, H., *and* Martin, C. J., Density and Solution Volume of Some Proteins, 279
Chlopin, G. W., *and* Wassiljewa, P. J., Method of Distinguishing Natural from Artificial Colouring Matters by Means of their Electrical Conductivity, 377

Christensen, H. R., Significance of the Estimation of the Reaction and Basicity of Soil, 462
Christiansen, J., Mett's Method for Determining the Activity of Pepsin and the Acidity-Maximum of Peptic Digestion, 29
————, J. A., *and* Lomholt, S., Estimation of Small Quantities of Mercury in Organic Substances, 515
Clark, A. H., *and* Wheeler, R. V., and with Platt, C. B., Volatile Constituents of Coal Part III., 511
Claude, G., Liquid Air, Oxygen, Nitrogen, *Review*, 600
Coade, M. E., *and* Werner, E. A., Estimation of Nitrites by Means of Thiocarbamide, and the Interaction of Nitrous Acid and Thiocarbamide in Presence of Acids of Different Strength, 440
Cocking, W. D., *and* Kettle, J. D., Estimation of Hypophosphites, 438
Coen, E., Stearin of Wool Fat, 37
Cohn, R., Lecithin Preparations and the Estimation of Lecithin, 205
Collins, W. D., *and* Skinner, W. W., Determination of Lithium, 168
Comey, A. M., *and* Holmes, F. B., Methods for the Determination of the Effective Strength of High Explosives, 378
Connell, W. B., *and* Francis C. K., Colorimetric Method for the Determination of Hydrocyanic Acid in Plants, with Special Reference to Kaffir Corn, 569
Constontino, A., Amino-Acids in Blood, 567
Contassot, A., Ebullioscope of High Accuracy, 482
Cornelius, W., New Separation of Chromium and Manganese, 474
Coste, J. H., Notes on Thermometry, 303
————, *and* Huntly, G. N., Determination of Moisture, 174
Cowell, C. F., *and* Bell, J. M., Methods for the Preparation of Neutral Solutions of Ammonium Citrate, 116
Crawford, S. L., Detection of Adulteration of Cider Vinegar with Distilled Vinegar, 565
Cribb, C., The Detection of Adulteration in Linseed Oil, 7
Cross, W. E., Investigations on Methods of Analysis of Cane Products, 415
Crown, H. A., *and* Porst, C. E. G., Polarimetric Method for the Determination of Starch in Paper, 380
Cumming, A. C., *and* Finlay, T. M., Estimation of Zinc as Zinc Ammonium Phosphate and Zinc Phosphate, 443
————, *and* Gilchrist, E., Electrolytic Determination of Copper in Solutions Containing Nitric Acid, 340
————, *and* Macleod, J., Estimation of Mercury as Metal by the Dry Method, 227
Curtman, L. J., *and* Mosher, M., Detection of Tin, 297
Czyhlarz, E. v., Fuchs, A., *and* Fürth, O. v., Analysis of Human Bile, 208

Daish, A. J., *and* Davis, W. A., Methods of Estimation of Carbohydrates, especially in Plant Extracts, 504
Damiens, A., *and* Lebeau, P., Analysis of Mixtures of Hydrogen with Methane, Ethane, and Propane, 121
————, Analysis of Mixtures of Hydrogen with Methane, Ethane, Propane, and Isobutane, 276
————, Estimation of Hydrocarbons of the Acetylene and Olefine Series in Mixtures of Gaseous Hydrocarbons, 158
Dapper, M., Estimation of Lactic Acid in Urine, 370
Davies, S. H., *and* Bainbridge, J. S., Essential Oil of Cocoa, 20
Davis, —, *and* Sadtler, —, Allen's Commercial Organic Analysis. Vol. VII., *Review*, 597

Davis, W. A., Apparatus for the Continuous Evaporation of Large Volumes of Liquid under Diminished Pressure, 591
———, Estimation of Potassium in Fertilisers, Soil Extracts, and Plant Ashes, 47
———, *and* Daish, A. J., Methods of Estimation of Carbohydrates, especially in Plant Extracts, 504
Dederichs, W., Separation of Chromium and Manganese, 474
Dejeanne, A., Gravimetric Estimation of Carbon Dioxide, 386
———, Method of Estimating Carbon Dioxide, 437
Demorest, D. J., Analysis of Alloys of Lead, Tin, Antimony, and Copper, 584
———, Determination of Chromium and Vanadium in Steels, 81
———, Determination of Zinc in Ores, 298
———, Electrolytic Estimation of Copper in Ores Containing Arsenic, Antimony, or Bismuth, 222
———, Thiocyanate-Permanganate Method for Copper in Ores, 223
Demoussy, E., *and* Maquenne, L., Determination of Respiratory Quotients, 108
———, Respiratory Quotients of Green Plants, 152
Denigès, G., Estimation of Traces of Free Bromine, 119
———, Preservation of Milk Samples for Analysis, 325
———, *and* Chelle, L., New Reagent for Bromine and Chlorine, 119
Dennis, L. M., *and* O'Brien, W. J., Determination of Phosphorus in Commercial Acetylene, 46
Deshayes, A., *and* Bourion, F., Quantitative Separation of Chromium and Aluminium: Analysis of Chromite, 473
———, Quantitative Separation of Iron and Chromium, 387
Devarda, A., *and* Weich, A., Morres's "Alizarol" Test for Milk, 460
Dewey, F. P., Gay-Lussac Method of Silver Determination, 228
Diakow, M., Use of Bomb Calorimeter for Elementary Analysis and Heat of Combustion, 533
Diebel, C., Ehrenberg, P., *and* Veckenstedt, H., Estimation of Organic Matter in Clays, 374
Dieckmann, T., *and* Hilpert, S., Separation of Arsenic and Tungsten, 117
Diedrichs, A., *and* Sprinkmeyer, H., Kapok Seeds and Kapok Oil, 467
Dittrich, M., Estimation of Small Quantities of Manganese and Chromium in Minerals and Rocks, 226
———, Estimation of Sulphur in Pyrites and Other Sulphides, 480
———, Estimation of Water in Silicate Minerals and Rocks, 47
Dodge, F. D., Essential Oil of Jamaica Ginger, 56
———, Estimation of Benzaldehyde and Oil of Bitter Almonds, 19
———, Oxidation Assay of Essential Oils, 69
Dodt, J., *and* Strigel, A., Estimation of Potassium as Perchlorate in Potash Fertilisers, 124
Dominicis, A. de, Gold Bromide Test for Semen, 29
Donau, J., Quantitative Manipulation of Small Precipitates, 302
Doolittle, R. E., Report of the Acting Chemist for 1912, U.S. Department of Agriculture, 131
Döring, T., Volumetric Estimation of Calcium, 518
Dreaper, W. P., Notes on Chemical Research, *Review*, 180
Duboux, —, Physico-Chemical Estimation of Sulphates, Tartaric Acid, Calcium, and Magnesium in Wine, 421
———, M., *and* Dutoit, P., Estimation of Tartaric, Succinic, and Malic Acids in Wine, 502
Dubrisay, R., New Physico-Chemical Volumetric Method, 294
Ducelliez, F., Method of Analysing Hypochlorites, 521
———, Titration of Chlorine Bleaches, 340

Dunbar, P. B., Determination of Malic and Tartaric Acids in the same Solution, 381
————, The Determination of Tartaric Acid, 380
Dunstan, A. E., *and* Strevens, J. F., Viscosity of Lubricating Oils, 36
Dupré, F. H., *and* Dupré, P. V., Constant Temperature Apparatus, 308
Durier, E., Estimation of Glycyrrhizin in Sweets and in Liquorice, 367
Duserre, C., *and* Chavan, P., Estimation of Phosphorus in Manures and Feeding-Stuffs, 563
Dutoit, P., *and* Duboux, M., Estimation of Tartaric, Succinic, and Malic Acids in Wine, 502
Dyer, B., A Simple Test for Differentiating between Cocoa Butter and "Green Butter," 203

Eastick, J. J., Ogilvie, J. P., *and* Lindfield, J. H., Estimation of Traces of Iron in Cane and Beet Sugar Factory and Refinery Products, 59
Eck, J. J. van, Use of Sodium Potassium Tartrate in the Estimation of Dissolved Oxygen in Water, 442
Eder, R., Microsublimation of Alkaloids under Reduced Pressure, 426
Edwards, J. D., *and* Hunter, W. H., Improved Apparatus for Determination of Carboxyl Groups in Organic Acid, 299
Egerton, A. C., Heat Test: 1. Guncotton; 2. Nitro-glycerine and Cordite; 3. Theoretical, 275
Ehrenberg, P., Diebel, C., *and* Veckenstedt, H., Estimation of Organic Matter in Clays, 374
Eibner, A., *and* Muggenthaler, H., Hexabromide Number of Fatty Oils, and the Estimation of Rape Oil in Raw Linseed Oils, 158
Einbeck, H., Occurrence of Succinic Acid in Meat Extract and Fresh Meat, 508
Ekeley, J. B., Distribution of the Ante-Mortem Administered Arsenic in the Human Body, 268
Elsden, A. V., *and* Stansfield, J. F., Estimation of Small Quantities of Lead, 440
Elsdon, G. D., Alcoholysis and the Composition of Cocoanut Oil, 8
————, *and* Hawley, H., The Detection of Adulteration in Linseed Oil, 3, 104
————, *and* Sutcliffe, J. A. L., Nitrates and Nitrites in Milk, 450
Enderli, M., *and* Stuckert, L., Bomb with Stirring Gear for the Measurement of Reaction; Velocity in a Heterogeneous System at High Pressures; New High-Pressure Reducing Valve, 590
Enz, K., Examination of Chloroform, 497
Esclangon, E., New Thermo-Regulator, 398
Euler, H., General Chemistry of the Enzymes, *Review*, 84
Evans, U. R., *and* Rideal, E. K., An Electro-chemical Indicator for Oxidising Agents, 353
Evers, N., The Detection of Adulteration in Linseed Oil, 7
————, The Estimation of Iron in *Syr. Ferri Phosph. Co.* and Other Pharmaceutical Preparations, 447

Fahrion, W., Estimation of Rosin in Blown Gils, 469
Feinberg, B. G., Quantitative Study of Some Aldehyde Reactions, 154
Feld, W., Estimation of Polythionate in Presence of Thiosulphate and Free Sulphurous Acid, 392
Fellenberg, T. von, Colour Reaction of Wine, 24
————, Estimation of Higher Alcohols in Wine, 23
————, Estimation of Quercitin in Wine, 267
————, Estimation of the Bromine Absorption of Wines, 266
————, Investigations on Lead Precipitates from Wines, Estimation of Pentoses and Methylpentoses in Grapes and Wines, 25

Ferguson, J. M., *and* Campion, A., Preparing Sections of Fractures of Steel for Microscopic Examination, 528
Ferre, L., *and* Mathieu, L., Estimation of Tartaric, Citric, Lactic, and Malic Acids in Musts, Wines, etc., 504
Fichter, F., *and* Müller, E., Volumetric Method for the Estimation of Tin by Means of Potassium Bromate, 229
Fiehe, J., *and* Stegmuller, P., Composition of Honey, 265
Fieldner, A. C., Accuracy and Limitations of Coal Analysis, 211
Fincke, H., Detection and Estimation of Formic Acid, 378
————, Method of Distinguishing Jamaica Rum from Artificial Rum, 368
Finlay, T. M., *and* Cumming, A. C., Estimation of Zinc as Zinc Ammonium Phosphate and Zinc Phosphate, 443
Finzi, C., *and* Rappuzzi, E., Rapid Method of Estimating Lead Peroxide in Minium, 343
Fischer, J., *and* Freundlich, H., Influence of Colloids on the Electrolytic Separation of Lead, 76
Florentin, D., Monohydrated Ferrous Sulphate and its Employment in Volumetric Analysis, 284
Fol, J. G., Estimation of the Viscosity of Rubber Solutions, 272
Folin, O., *and* Bosworth, A. W., Application of Folin's Method for the Determination of Ammonia to Fertilisers, 381
————, *and* Macallum, A. B., Junr., Colorimetric Method for the Estimation of Uric Acid in Urine, 154
Forbes, G. S., *and* Bartlett, E. P., Electrometric Titration of Dichromate Ion with Ferrous Iron, 585
————, W. R., Estimation of Solubilities, 84
Formenti, C., Copper in Cocoa and Chocolate, 145
Fortini, V., Calorimetric Detection and Estimation of Magnesium Oxide in Magnesium Carbonate, Asbestos Mixtures, etc., 76
————, *and* Ceccherelli, A., Testing Parchment Papers, 161
Francis, C. K., *and* Connell, W. B., Colorimetric Method for the Determination of Hydrocyanic Acid in Plants, with Special Reference to Kaffir Corn, 569
————, F., *and* Geake, F. H., New Method for the Determination of the Concentration of Hydroxyl Ions, 522
Franck, H. H., Simple Modification of Skita's Hydrogenising Apparatus, 484
Frank, G., *and* Moeser, L., Estimation of Phosphoric Acid, 344
Frankforter, G. B., *and* Frary, F. C., Method for Estimating Water in Alcohol. Equilibria in Systems Containing Alcohols, Water, and Potassium Fluoride, 425
Frary, F. C., *and* Frankforter, G. B., Method for Estimating Water in Alcohol. Equilibria in Systems Containing Alcohols, Water, and Potassium Fluoride, 425
————, *and* Mastin, M. G., Estimation of Zinc in Treated Ties (Railway Sleepers), 589
Frerichs, G., Testing Ether, 560
————, K., *and* Behre, A., Composition of Vermouth Wines, 267
Fresenius, R., Development of Electro-Analysis, 341
————, W., Present Position and Importance of Analytical Chemistry, 83
————, *and* Grünhut, L., Detection of Citric Acid in Wines, 55
Freundler, P., Estimation of Phosphorus in Lecithin, 58
Freundlich, H., *and* Fischer, J., Influence of Colloids on the Electrolytic Separation of Lead, 76
Friedrichs, F., Absorption Apparatus for Use in Nitrogen Estimations by Dumas' Method, 234
————, Extraction with Liquefied Gases, 233

Friese, W., Apparatus for Recovery of Evaporated Organic Solvents, 398
Fritsch, C., *and* Rogée, H., Estimation of Chlorine in Blood, 461
Frye, C. C., McGowan, G., *and* Kershaw, G. B., Royal Commission on Sewage Disposal: Appendix to Eighth Report, March, 1913, 539
Fry, W. H., Mineralogical Analysis of Soils, 125
Fuchs, A., Fürth, O. v., *and* Czyhlarz, E. v., Analysis of Human Bile, 208
——, D., *and* Abderhalden, E., Accuracy of the Estimation of *l*-Tyrosine in Proteins, 219
Fuller, H. C., The Qualitative Analysis of Medicinal Preparations, *Review*, 403
Funk, A., *and* Autenrieth, W., Colorimetric Methods for Water Analysis by the Use of Autenreith-Kœnigsberger's Colorimeter, 179
——, C., Attempt to Estimate the Vitamine Fraction in Milk, 269
Fürth, O. v., Czyhlarz, E. v., *and* Fuchs, A., Analysis of Human Bile, 208

Gadais, L., *and* Gadais, J., Goldenberg Method (for Commercial Tartrates), 1898 and 1907, 435
Gaither, E. W., Comparison of Some Qualitative and Quantitative Methods for Carbonates in Soils, 163
Gardner, W. M., *and* Lloyd L. L., Estimation of Mercury in Textile Materials, 63
Garratt, F., Colorimetric Method for Determination of Chromium in Steel, 285
Gartenmeister, R., Cathodic Estimation of Lead and Analysis of Lead Alloys, 582
Gastaldi, E., Modification of Halphen's Test for Cottonseed Oil, 68
——, *and* Pertusi, C., Detection of Hydrocyanic Acid, 332
Gaze, R., Estimation of Platinum by Precipitation as Sulphide, 173
Geake, F. H., *and* Francis, F., New Method for the Determination of the Concentration of Hydroxyl Ions, 522
Gemmell, W., Analysis of Copper-Tin Alloys, 388
Gibbs, H. D., Interference of Hydrogen Peroxide with the Milk Tests for Formaldehyde, 21
Gildemeister, E., *and* Hoffmann, Fr., The Volatile Oils, *Review*, 547
Gile, P. L., *and* Ageton, C. N., Significance of the Lime-Magnesia Ratio in Soil Analyses, 126
Gill, H. W., The Recovery of Iodine from Residues, 409
Gilpin, J. E., *and* Schneeberger, P., Fractionation of California Petroleum by Diffusion through Fuller's Earth, 469
Glenk, K., König, J., *and* Hasenbaümer, J., Application of Dialysis and Estimation of Oxidising Power as Aids in Judging Soils, 369
Goldberg, P., Estimation of Caoutchouc in Rubber Wares, Particularly Insulated Wires, 111
Golding, J., The Bacterial Testing of Disinfectants: A Practical Criticism, 198
——, The Composition of Milk, 254
Golodetz, A., New Apparatus for Quantitative Dialysis, 232
Gooch, F. A., *and* Blumenthal, P. L., Application of Selenic Acid to the Estimation of Bromide in Presence of Chloride, 227
——, Iodic Acid Method of Estimating Bromine in Halogon Salts, 166
——, *and* Burdick, W. L., Electro-Analysis with Light Platinum Electrodes, 43
——, *and* Hill, D. U., Purification of the Precipitate Obtained in Estimating Barium as Sulphate, 339
–, F. D., Methods in Chemical Analysis Originated or Developed in the Kent Chemical Laboratory of Yale University, *Review*, 489

Gowing-Scopes, L., Examination of the Oils from *Manihot Ceara* and *Funtumia Elastica* and a Comparison of their Properties with those of Linseed and *Hevea* Oils, 262

——————, The Estimation of Citric Acid in the Presence of Certain Other Acids, 12

Graaff, W. C. de, *and* Schaap, A., Estimation of Proteins in Milk by Aldehyde Titration, 266

Grassi, L., *and* Bellucci, J., Use of " Cupferron " in the Separation of Titanium from Aluminium, 390

Graves, S. S., *and* Kober, P. A., Quantitative Ammonia Distillation by Aëration, III., 580

Greaves, J, E., Factors Influencing the Estimation of Arsenic in Soils, 221

————, *and* Stewart, R., Influence of Chlorine on Determination of Nitrates by Phenol Disulphonic Acid Method, 339

Greef, A., Volumetric Estimation of Fluorine, 521

Grewing, B., Detection of Potassium Bichromate in Milk, 563

Griebal, C., Reaction of Peru Balsam, 58

Griebel, G., Yoghurt and Yoghurt Preparations, 31

Grignard, V., Mechanical Agitator, 590

Grimaldi, C., Influence of Antiseptic Agents on the Reichert-Meissl Value of Fats, 68

————, *and* Prussia, L., Maumené Figure of Oil of Turpentine, 338

Grimbert, L., Estimation of Reducing Sugars by Lehmann's Method, 219

————, *and* Laudat, M., Estimation of Lipoids in Blood-Serum, 28

Grimme, C., Apparatus for Use in the Estimation of Starch by Ewers' Process, 445

————, Colour Reactions of Hardened Whale Oil, 433

————, Hardened Marine Animal Oils, 373

————, Oil of Manketti Nuts, 105

Grob, W., *and* Bosshard, E., Analysis of Hydrosulphites, 284

——————, New Process for Titration of Sulphurous Acid and Sulphites in Presence of Thiosulphates, 297

Grosser, P., Effect of Boiling on the Physico-Chemical Behaviour of Human Milk, Cow's Milk, and Butter-milk, 205

Groszfeld, J., *and* König, J., Fish Roe as Human Food, 508

Grünberg, P., Blacher, C., *and* Kissa, M., Use of Potassium Palmitate in Water Analysis, 127

Grünhut, L., *and* Fresenius, W., Detection of Citric Acid in Wines, 55

Grünwald, B., Examination of Portland Cement, 437

Grützner, R., *and* Blum, F., Estimation of Iodine in Organic Substances, 427

Gruzewska, Z., *and* Bierry, H., Estimation of Glycogen in Liver, 63

Guareschi, I., Characteristic Reaction of Bromine, 518

Characteristic Reaction of Free Bromine, 118

——, R., Detection of Saccharin in Vermouth, 22

Gude, K., *and* Pinoff, E., Estimation of Pentoses in Presence of Other Sugars by Means of the Spectroscope, 333

Guérin, G., Estimation of Morphine in Opium and Opium Preparations, 150

Guggiari, P. S., *and* Litterscheid, F. M., Estimation of Active Oxygen in Perborates and in Washing Powders Containing Perborates, 441

Guillanden, A., Pritz, W. B., *and* Withrow, J. R., Detection of Traces of Copper, 167

Guiterman, K. S., *and* Treadwell, W. D., Separation of Cadmium from Zinc, 383

Haas, F., Colorimetric Estimation of Small Quantities of Manganese in Water, 225

Halberkahn, J., Detection and Differentiation of Poisonous and Non-Poisonous Saponins by Hæmolysis, 151

Hall, S. G., *and* Harvey, A. J., Estimation of Glyceryl Acetate in Essential Oils, 148

Halle, W., Loewentstein, E., *and* Pribram, E., Colour Reactions of Triketohydrindene Hydrate (Ninhydrin), 579
Halverson, J. O., Modified Babcock Test for Fat in Sweetened Ice Cream, 323
Hammer, B. W., *and* Johnson, A. R., New Designs for Specific Heat Apparatus, 484
Hammerschmidt, —, Acid-Butyrometric Method for the Estimation of Fat in Cheese and Milk Products, 145
Hanuš, J., Estimation of Copper by Means of Sodium Hypophosphite, 519
Harding, E. P., *and* Johnson, E., Apparatus and Method for Determining Hydrogen Sulphide in Illuminating Gas, 396, 593
——————, *and* Nye, L. L., Rapid Control Method for the Determination of Oil in Grains, 57
——————, *and* Parkin, G., Rapid and Accurate Gravimetric Method for Determining Milk-Fat in Evaporated Milk and Milk Powders, 147
——————————————, Rapid and Accurate Method for Determining Fat in Ice Creams, Cereals, and Chocolates, 558
Hardisty, R. H. M., *and* Ruttan, R. F., New Reagent for the Detection of Traces of Blood, 422
Harrison, E. F., and Self, P. A. W., Analytical Constants of Extract of Male Fern, 429
Hart, E. B., *and* Woo, K. J., Simple Method for the Determination of Carbon in Organic Materials, 465
Hartwagner, F., Interaction between Formaldehyde and Bismuth or Copper Compounds in the Presence of Alkali, 66
Harvey, A. J., *and* Hall, S. G., Estimation of Glyceryl Acetate in Essential Oils, 148
————, E. N., Indicator Method for the Estimation of the Permeability of Cells by Alkalis, 327
Haschek, E., *and* Hönigschmid, O., Purity of the International Radium Standard, 178
Hasenbaümer, J., König, J., *and* Glenk, K., Application of Dialysis and Estimation of Oxidising Power as Aids in Judging Soils, 369
Hatschek, E., An Introduction to the Physics and Chemistry of Colloids, *Review*, 352
Hatta, Z., I. Bang's Method of Sugar Estimation, 435
Hausmann, J., Detection of Urobilin, 463
Hawley, H., *and* Elsdon, G. D., The Detection of Adulteration in Linseed Oil, 3, 104
————, L. F., Wood Turpentines—their Analysis, Refining, and Composition, 39
Haydin, S. von, Estimation of Water in Vegetables by Hoffmann's Apparatus, 177
Heap, H., Action of Various Waters on Lead, 530
Heermann, P., Estimation of Small Quantities of Wool in Cotton Goods, 510
Hehner, O., Constant Temperature Apparatus, 311
——————, Examination of the Oils from *Manihot Ceara* and *Funtumia Elastica*, and a Comparison of their Properties with those of Linseed and *Hevea* Oils, 263
Heide, C. von der, Estimation of Sulphate in Wines, 369
——————, *and* Kartschmar, M, Estimation of Chlorides in Wine 502
Heim, F., *and* Marquis, R., Estimation of Caoutchouc in Crude Rubber, 71
Heintz, W., *and* Limprich, R., Detection and Estimation of Salicylic Acid in Fruit Juices, 420
Heinz, R., Physiological Standardisation of Digitalis Preparations, 462
Heinzelmann, G., Valuation of Fusel Oil, 66
Hempel, W., Opening up Silicates, 125
Henderson, J. B., *and* Meston, L. A., Freezing Point of Milk, 368
Henri, V., *and* Bielecki, J., Quantitative Study of the Absorption of Ultra-Violet Rays by the Fatty Acids and their Isomeric Esters, 113
Henrich, J., Theorien der Organischen Chemie. Zugleich Zweite Auflage der Neueren Theoretischen Anschauungen auf dem Gebiete der Organischen Chemie, *Review*, 601

Hepp, K., Messger, O., *and* Jesser, H., Composition of Milk from Cows Suffering from Foot and Mouth Disease, 328
Herles, F., Estimation of Uric Acid in Urine, 65
Hesse, E., Bacteriological Methods of Water Analysis, with Special Reference to the Use of the Berkefeld Filter, 574
Hetper, J., Estimation of Methyl Alcohol in Spirits, 56
——— Estimation of Small Quantities of Methyl and Ethyl Alcohols in Aqueous Solutions, 573
Hevesy, G. v., *and* Paneth, F., Solubility of Lead Sulphide and Lead Chromate, 476
Heyl, G., *and* Kneip, P., Microscopical Detection of Embelic Acid, 559
Hicks, E. F., Shellac Analysis and the Detection of Small Amounts of Colophony in Shellac, 38
Higgins, W. F., Methods and Apparatus Used in Petroleum Testing. Part II.: Viscosimetry, 397
Hill, C. A., Extract of Male Fern, 428
———, D. U., *and* Gooch, F. A., Purification of the Precipitate Obtained in Estimating Barium as Sulphate, 339
Hilpert, S., Estimation of Carbon by Combustion with Moist Oxygen, 271
———, *and* Dieckmann, J., Separation of Arsenic and Tungsten, 117
Hinard, G., Alteration of Milk Treated with Potassium Dichromate, 325
Hinks, E., The Detection and Estimation of Benzoic Acid in Milk and Cream, 555
Hinman, J. J., Junr., Estimation of Total Formaldehyde in Fumigators and Commercial Solutions, 497
Hinrichsen, F. W., New German Regulations for the Official Examination of Inks, 213
———, Report on the Present State of Rubber Analysis, 336
Hirschberg, E., Estimation of Small Amounts of Glucose in Urine, 463
Hodgson, T. R., Note on Coffee and Chicory, 454
Hoepfner, W., *and* Burmeister, H., Examination of Tung Oil, 116
Hoffmann, Fr., *and* Gildemeister, E., The Volatile Oils, *Review*, 547
———, K. A., Osmium Tetroxide as an Oxygen-Carrier and the Rendering Active of Chlorate Solutions, 78
Hofsäss, M., Apparatus for Determining the Density and Internal Friction of Gases, 591
Holloway, G. T., Notes on Valuation of Ores and Minerals, and on Metallurgical Calculations, 171
Holmes, A., *and* Adams, M., Pine-Nut Oil, 275
———, F. B., *and* Comey, A. M., Methods for the Determination of the Effective Strength of High Explosives, 378
Homer, A., Colour Reactions of Certain Indole Derivatives and their Significance with Regard to the Glyoxylic Reaction, 276
Hönigschmid, O., *and* Haschek, E., Purity of the International Radium Standard, 173
Hooker, A. H., Chloride of Lime in Sanitation, *Review*, 405
Houston, A. C., Metropolitan Water Board: Seventh Annual Report, March 31, 1913, 487
———, Search for Pathogenic Microbes in "Raw" River Water and in Crude Sewage, 370
Hugel, E., *and* Normann, W., Analysis of Hardened Oils, 432
Hulbert, E. C., Improved Apparatus for Testing the Jelly-Strength of Glues, 231
Hunter, W. H., *and* Edwards, J. D., Improved Apparatus for Determination of Carboxyl Groups in Organic Acids, 299
Huntly, G. N., *and* Coste, J. H., Determination of Moisture, 174
Huybrechts, M., *and* Joassart, N., Estimation of Manganese in Castings and Steels by the Method of G. von Knorre, 286

Hyde, A. L., Quantitative Separation of Nitrosubstitution Compounds from Nitroglycerine, 513
——————, Unusual Case of Specific Gravity, 50

Ibbotson, F., *and* Aitchison, L., Determination of Copper in White Metal Alloys, 222
Imbert, H., *and* Juillet, A., Black Mustard Powder, 500
Inouye, K., Detection of Histidine, 114
Ishihara, H., Estimation of Lactic Acid in Urine, 328
Iwanow, W. N., Detection of Nitrate in Presence of Nitrite, 170
————, W., Estimation of Free Acid and Base in Aluminium Sulphate, 283
————, W. N., Qualitative Detection of Nitric Acid in Sulphuric Anhydride, 78

Jacob, W., *and* Müller, E., Action of Persulphate on Iodate, and Estimation of Periodate in Presence of Persulphate, 479
Jacobs, W. A., *and* Allen, J. C., Estimation of Water in Petroleum and its Products, 165
Jahnson-Blohm, G., Influence of Ammonium Sulphate on the Polarimetric Estimation of Lactose, 217
Jakob, W., Precipitation of Aluminium Hydroxide, and Separation of Aluminium and Chromium, 517
Jalade, —, Estimation of Free Sulphur in Leather, 467
Jámbor, J., Differentiation of Natural and Artificial Barium Sulphate, 517
James, C., *and* Smith, T. O., Some Quantitative Separations of Neodymium, 343
Jannasch, P., *and* Leiste, R., Quantitative Volatilisation and Separation of Phosphoric Acid from the Metals of the Copper Group, and from Aluminium, Tin, and the Alkali Metals, 525
Jarvinen, K. K., Estimation of Sugar (Dextrose) in Urine, 65
Jensen, H. R., Analysis of Peru Balsam, 150
Jesser, H., Mesger, O., *and* Hepp, K., Composition of Milk from Cows Suffering with Foot and Mouth Disease, 328
Joassart, N., *and* Huybrechts, M., Estimation of Manganese in Castings and Steels by the Method of G. von Knorre, 286
John, W. E. von, Analysis of Aluminium Alloys, 220
————————, Estimation of Total Carbon in Steel and Ferro-Alloys by Combustion in Oxygen under Pressure, 285
Johnson, A. R., *and* Hammer, B. W., New Designs for Specific Heat Apparatus, 484
————, C. M., Determination of Phosphorus in Ferrotungsten, Metallic Tungsten Powder, Tungsten Oxide, and Tungstic Acid, by Direct Solution, 290
————, E., *and* Harding, E. P., Apparatus and Method for Determining Hydrogen Sulphide in Illuminating Gas, 396, 593
Johnston, J., *and* Adams, L. H., Improved Pyknometer for Density of Solids, 130
Jolles, A., Detection of Pentoses in Diabetic Urine, 64
—————, New Indican Reaction, 508, 578
Joly, J., Apophorometer, 302
Jona, T., Peroxydase Reaction of Milk. (Influence of Preservatives, etc.), 325
Joseph, A. F., *and* Rae, W. N., Notes on Alcoholometry, 508
Juillet, A., *and* Imbert, H., Black Mustard Powder, 500

Kafka, E., Quantitative Precipitation of Tungstic Acid by Means of Aromatic Amines, 529
Kalusky, L., Simple Extraction Apparatus, 49
Karas, J., Estimation of Saccharin in Foods, 326
Kardos, M., *and* Schiller, W., Improved Apparatus for Extracting Finely divided Solids with Volatile Solvents, 444
Kartschmar, M., *and* Von der Heide, C., Estimation of Chlorides in Wine, 502

Kaschinsky, P., Hydrolytic Action a Source of Error in the Estimation of Iodine and Bromine in Mineral Waters, etc., 532
Kast, H., Estimation of Brisance and of Velocity of Detonation, 464
Kay, S. A., *and* Walker J., Acidity and Alkalinity of Natural Waters, 40
Kedesdy, E., Detection of Molybdenum, 523
Kempf, R., Apparatus for Extracting Liquids Decomposed by Heat, 444
Kershaw, G. B., McGowan, G., *and* Frye, C. C., Royal Commission on Sewage Disposal: Appendix to Eighth Report, March, 1913, 539
Kettle, J. D., *and* Cocking, W. D., Estimation of Hypophosphites, 438
Kickton, A., *and* Murdfield, R., Composition of Port Wine, 368
Kingzett, C. T., *and* Woodcock, R. C., The Bacterial Testing of Disinfectants: A Practical Criticism, 190
Kirchhof, F., Experiments on the Direct Estimation of Caoutchouc by Titration with Bromine, 272
Kissa, M., Blacher, C., *and* Grünberg, P., Use of Potassium Palmitate in Water Analysis, 127
Klassert, M., Studies on Caoutchouc Resins, 465
Klemenc, A., Volumetric Method for Methoxyl and Ethoxyl Estimations, 430
Klinger, G., Gasometric Estimation of Nitric Oxide and Oxygen, Depending on the Formation of Nitrous Anhydride, 391
——, *and* Baudisch, O., New Gasometric Method for the Estimation of Nitric Oxide: I., 45
Knapp, A. W., Note on "Hardened" Oils, 102
Kneip, P., *and* Heyl, G., Microscopic Detection of Embelic Acid, 559
Kober, P. A., Nephelometric Determination of Proteins; Casein, Globulin, and Albumin in Milk, 571
——, *and* Graves, S. S., Quantitative Ammonia Distillation by Aëration, III., 580
——, *and* Sugiura, K., Micro-Chemical Method for the Estimation of α- and β-Amino Acids and Certain Derivatives in Proteolysed Substances, Blood, and Urine, 567
Koch, B., Analysis of Brass, 436
Koehler, —, *and* Marqueyrol, —, Estimation of Nitric Oxide, 171
Koelsch, H., Rapid Estimation of Copper in Burnt Pyrites, 389
Kohn-Abrest, E., Action of "Activated" Aluminium on Alkaloid Extracts and its Employment in Toxicology. 61
——, Use of Amalgamated Aluminium in the Estimation of Tannin, 516
——, Rochas, —, *and* Rivera-Maltes, —, Action of "Activated" Aluminium on Alkaloid Extracts and its Use in Toxicology, 268
Komarowsky, A., Sensitive Reaction of Molybdenum, 478
Konek-Norwall, F. v., Calorimetric Ash Estimations, 582
König, F., *and* Matthes, H., Estimation of Crude Fibre and Cellulose, 415
——, H., Estimation of Cobalt and Uranium in Steel, 527
——, J., Non-Nitrogenous Extractive Matters in Fodder and Foodstuffs, 560
——, J., *and* Groszfeld, Fish Roe as Human Food, 508
——, Hasenbaümer, J., *and* Glenk, K., Application of Dialysis and Estimation of Oxidising Power as Aids in Judging Soils, 369
Kooper, W. L., Estimation of Fat in Cheese by the Acid-Butyrometric Method without the Use of Amyl Alcohol, 146
Kopaczewski, W., Dialysing Apparatus, 394
Körösy, K. V., Micro-Colorimeter for Measuring Heat Evolved by Bacteria, 421
Korreng, E., Preparation of Sections of Crystalline Substances, 483
Korsakoff, M., Methods for the Estimation of Saponin, 22

Kreis, H., *and* Roth, E., Fractional Precipitation of Fatty Acid Mixtures (Detection of Rape Oil), 114
——————, Solidified (Hydrogenised) Oils and the Detection of Arachidic Acid, 160
Kremann, R., The Application of Physico-Chemical Theory, *Review*, 599
Kretschmer, E., Estimation of Lactose in Milk by Precipitation with Ammonium Sulphate, 418
——————, Titration of Uric Acid in Urine after Silver Precipitation, 329
Kries, H., Detection of Rape Oil in Olive Oil, 434
Krogh, M., Estimation of Urea in Urine by Sodium Hypobromite, 269
Kropat, K., Estimation of Iron in *Extractum Ferri Pomati*, 149
Krüger, A., Estimation of Benzoic Acid in Meats, 414
Kruysse, P. J., Quinine Assay, 59
Kühl, H., Disinfectant Value of Mercuric Hydroxycyanide, 461
Kullgren, C., Estimation of Sulphurous Acid in Air, 127
Kurtenacker, A., Volumetric Estimation of Chromium in Presence of Iron, 387
Kurzmann, I., Multiviscosimeter, 178

Labbé, H., *and* Maguiss, R., Studies of the Conditions of the Precipitation of Albumin by Picric Acid, 329
Lamb, M. C., The Analysis of Various East-Indian Tanned Hides, 363
Lambris, G., Estimation of Water in Volatile Products of Destructive Distillation, 348
Lander, G. D., *and* Winter, H. W., Antipyrin in Toxicological Analysis, 97
Langheld, K., *and* Zeileis, A., Analysis of Mixtures of the Lower Fatty Acids, 273
Lantenois, M., Estimation of Carbon Tetriodide in the Presence of Iodoform, 373
Larsen, C., *and* White, W., Dairy Technology, *Review*, 544
Larsson, K. O., Estimation of Chlorides in Urine and Blood, 270
Laudat, M., *and* Grimbert, L., Estimation of Lipoids in Blood-Serum, 28
Leach, A. E., Food Inspection and Analysis, *Review*, 490
Leather, J. W., Determination of the Permeability of Soils to Water, 49
Lebeau, P., *and* Damiens, A., Analysis of Mixtures of Hydrogen with Methane, Ethane, and Propane, 121
——————, Analysis of Mixtures of Hydrogen with Methane, Ethane, Propane, and Isobutane, 276
——————, Estimation of Hydrocarbons of the Acetylene and Olefine Series in Mixtures of Gaseous Hydrocarbons, 158
Leclère, A., Detection of Nitrous Acid in the Presence of Nitric Acid, 585
——————, Reduction of Ferric Salts, and their Titration with Potassium Permanganate, 390
Leduc, E., *and* Chenu, G., Matériaux de Gros-Ouvre, *Review*, 181
Lee, J. E., *and* Mathers, —, Determination of Hydrogen, Nitrogen, and Methane in Gas by Combustion in a Quartz Tube, 341
—, R. E., Uhlinger, R. H., *and* Amon, F. O., Qualitative Analysis of the Zinc Group, 349
Leech, P. N., Colour Reaction of Hypochlorites with Methylaniline and Ethylaniline, 476
Lehmann, F., Direct Iron Estimation in *Ferri et Quininæ Citras* and *Extractum Ferri Pomatum*, 562
——————, Estimation of Arsenic in Blood and Urine, 106
Leiste, R., *and* Jannasch, P., Quantitative Volatilisation and Separation of Phosphoric Acid from the Metals of the Copper Group, and from Aluminium, Tin, and the Alkali Metals, 525
Lemeland, P., Method for the Direct Polarimetric Determination of Sucrose in Presence of Certain Reducing Sugars, 71

Lenher, V., Volumetric Determination of Gold, 390
Lenz, W., Microchemical Reagents, 83
Lesser, E. J., Sources of Error in the Estimation of Sugar in the Blood of Frog and Tortoise, 463
Lessing, R., An Electrochemical Indicator for Oxidising Agents, 362
Leuze, W., *and* Pfister, R., Table of Calculated Dry Substance of Milk by the Fleischmann Formula, 324
Levallois, F., *and* de Vilmorin, P. L., Hydrolysis of Lævulosans and its Application for Analytical Purposes, 429
Levy, L. A., Apparatus for the Examination of Mine Air, 128
Lewin, L., Colour Reaction of Proteins, 380
Lewis, R. H., *and* Reeve, C. S., Application of the Dimethyl Sulphate Test for Determining Small Amounts of Petroleum or Asphalt Products in Tars, 277
Lifschütz, I., Study of the Oxydation Products of Cholesterol, 209
Lifschutz, J., Estimation of Cholesterol Bodies in Presence of Each Other, 466
Limprich, R., *and* Bömer, A., Nomenclature of Mixed Glycerides and the Synthetic Preparation of α-Distearin and β-Palmitodistearin, 214
————————, Polenske "Difference Value" for the Detection of Certain Animal Fats and its Theoretical Basis, 204
————, *and* Heintz, W., Detection and Estimation of Salicylic Acid in Fruit Juices, 420
Linder, E., Valuation of Hydrated Oxide of Iron by Means of the "Sulphuretted Hydrogen Burette," 522
Lindet, L., Solubility of Milk Proteins in the Constituents of the Serum, 570
Lindfield, J. H., Eastick, J. J., *and* Ogilvie, J. P., Estimation of Traces of Iron in Cane and Beet Sugar Factory and Refinery Products, 59
Lipman, C. B., *and* Pressey, H. F., Contribution to Methods of Determining Nitrogen in Humus, 163
Litterscheid, F. M., Detection of [Commercial] Invert Sugar by Means of β-Naphthol, 217
——————, *and* Guggiari, P. S., Estimation of Active Oxygen in Perborates and in Washing Powders Containing Perborates, 441
Lloyd, L. L., *and* Gardner, W. M., Estimation of Mercury in Textile Materials, 63
Loewentstein, E., Halle, W., *and* Pribram, E., Colour Reactions of Triketohydrindene, Hydrate (Ninhydrin), 579
Lomholt, S., *and* Christiansen, J. A., Estimation of Small Quantities of Mercury in Organic Substances, 515
Lomschakow, A., Gas Analysis Apparatus, 594
Lowenstein, A., *and* Beolio, L., Experiments on the "Cold Test" of Oil, 34
Lucas, H. J., Estimation of Nitrobenzene in Earthnut Oil, 457
Lüers, H., Detection of Earthnut (Arachis) Oil in Olive Oil by the Franz-Adler Method, 58

Macallum, A. B., Junr., *and* Folin, O., Colorimetric Method for the Estimation of Uric Acid in Urine, 154
Macara, T., Combined Estimation of Boric Acid and Fat in Cream, 142
MacDougall, F. H., Estimation of Potassium by Cobaltinitrite Method, 81
Macfadden, A. W. J., Report on the Work of Inspectors of Foods for the Year 1911-1912, 131
Mackey, W. McD., Rapid Method of Testing Suction Gas Fuels for Liability to clog Gas Engine Valves, 332
Macleod, J., *and* Cumming, A. C., Estimation of Mercury as Metal by the Dry Method, 227
Mäder, H., *and* Rupp, E., Estimation of Hydroxylamine, 437

Maguiss, R., *and* Labbé, H., Studies of the Conditions of the Precipitation of Albumin by Picric Acid, 329
Mai, C., *and* Rheinberger, E., Estimation of Water in Cheese, 146
Malacarne, M., Colour Reactions of Oils with Certain Phenols in the Presence of Nitric Acid, 433
Malatesta, G., and Nola, E. D., Detection of Gold and Platinum, 476
Malenfant, R., Estimation of Casein and Lactose in Milk, 21
Malvezin, P., Accurate Estimation of the Dry Extract of Wines and Fermented Liquors, 566
——————., New Reaction for the Detection of Aniline Dyestuffs (Magenta) in Foods and Especially in Wines, 322
Mannhardt, H., Oxygen Absorption Test for Linseed Oil, 159
Maquenne, L., *and* Demoussy, E., Determination of Respiratory Quotients, 108
——————————————., Respiratory Quotients of Green Plants, 152
Marcus, E., *and* Biltz, W., Titration of Phosphoric and Boric Acids, 124
Marcusson, J., Estimation of Soaps in Lubricating Greases, 162
——————, Quantitative Estimation of Natural, in Presence of Artificial Asphaltum, 156
——————, *and* Schilling H., Differentiation of Animal and Vegetable Fats, 458
——————, *and* Schwarz, F., Examination of Steam Turbine Oils, 468
——————, *and* von Skopnik, A., Distilled Wool Fat, 106
Marino, L., *and* Pellegrini, A., Volumetric Estimation of Hypophosphorous Acid and Hypophosphites, 342
Marqueyrol, —, *and* Koehler, —, Estimation of Nitric Oxide, 171
Marquis, R., *and* Heim, F., Estimation of Caoutchouc in Crude Rubber, 511
Marrs, L. E., *and* Metzger, F. J., Volumetric Determination of Manganese in Rocks, Slags, Ores, and Spiegels, 170
Marsh, H. L., *and* Welker, W. H., Aluminium Hydroxide as a Protein Precipitating Reagent in the Estimation of Lactose in Milk, 418
Marshall, J., *and* Welker, W. H., Precipitation of Enzymes from their Solutions by Means of Moist Aluminium Hydroxide, 423
Martin, C. J., *and* Chick, H., Density and Solution Volume of Some Proteins, 279
———, G., *and* Others, Industrial and Manufacturing Chemistry: Organic, *Review*, 399
Martindale, W. H., Digitalis Assay, *Review*, 237
Martini, C., Detection of Saffron in Confectionery, 206
Massy, R., Detection of Petroleum Products in Oil of Turpentine by Means of the the Maumené Test, 70
Masters, H., *and* Smith, H. L., Behaviour of Calcium and Magnesium Salts with Soap Solutions, and the Determination of Hardness of Water, 436
Mastin, M. G., *and* Frary, F. C., Estimation of Zinc in Treated Ties (Railway Sleepers), 589
Mathers, —, *and* Lee, J. E., Determination of Hydrogen, Nitrogen, and Methane in Gas by Combustion in a Quartz Tube, 341
Mathieu, L., *and* Ferre, L., Estimation of Tartaric, Citric, Lactic, and Malic Acids in Musts, Wines, etc., 504
Matthes, H., *and* König, F., Estimation of Crude Fibre and Cellulose, 415
——————, *and* Streicher, L., Waxes and Resins of Kapok and Akon Fibres, 580
Matthewson, W. E., Identification of Small Amounts of Dyestuffs by Oxidation with Bromine, 376
————————, Quantitative Separation and Determination of Subsidiary Dyes in the Permitted Food Colours, 375
————————, Quantitative Separation of Mixtures of Certain Acid Coal-Tar Dyes, 112

Maurantonio, L., *and* Settim, L., Detection of Vegetable Oils in Margarine, 21

Mayer, O., Detection of Albumin in Urine, 573

———, P., Residual Reduction of Blood, 327

McBride, R. S., *and* Weaver, E. R., Estimation of Sulphate in Ammonium Sulphate Solution, with Special Reference to the Testing of Illuminating Gas, 382

McCabe, C. R., Colorimetric Method for Titanium in Iron and Steel, 588

———, Estimation of Vanadium in Steel by the Hydrogen Peroxide Colour Method, 589

McCandlish, D., and Wilson, J. A., Estimation of Alkali Sulphides, 393

McClintic, T. B., Method of Standardising Disinfectants with and without Organic Matter, 422

McGowan, G., Frye, C. C., *and* Kershaw, G. B., Royal Commission on Sewage Disposal: Appendix to Eighth Report, March; 1913, 539

McMillen, R. H., Application of Electric Resistance Furnace to Determination of Oxygen in Iron and Steel, 168

Mecklenburg, W., Separation of Phosphoric Acid by Means of Stannic Acid in Qualitative Analysis, 292

Medinger, P., Automatic Pipette, 534

Meeker, G. H., Prevention of Emulsification in Extractions by Immiscible Solvents, 48

Meimberg, —, *and* Winzer, P., Separation of Niobium and Tantalum by Means of Potassium Chloride in Hydrofluoric Acid Solution, 226

Meissner, W., Comparison of Redwood's and Engler's Viscosimeters, 398

Meldrum, R., Solidifying and Melting-Points of Mixtures of Stearic and Oleic Acids, 579

Melikow, P., Sensitive Reaction for Molybdic Acid, 43

Merriam, E. S., *and* Birchby, J. A., Methods for the Estimation of Natural Gas for the Production of Gasoline, 577

Mesger, O., Jesser, H., *and* Hepp K., Composition of Milk from Cows Suffering from Foot and Mouth Disease, 328

Meston, L. A., *and* Henderson, J. B., Freezing-Point of Milk, 368

Metzger, F. J., *and* Marrs, L. E., Volumetric Determination of Manganese in Rocks, Slags, Ores, and Spiegels, 170

Meurice, R., Action of an Excess of a Soluble Ferrocyanide on Solutions of Zinc, Copper and Nickel, and a Volumetric Method for the Estimation of Nickel, 524

Meyer, H., *and* Beer, R., Composition of Arachis Oil, 514

———, *and* Soyka, W., Candelilla Wax, 514

Meyerfeld, J., Estimation of Methyl and Ethyl Alcohols in the Presence of each other, 330

Michel, F., Direct Estimation of Water in Foods, etc., by Distillation, 207

Michiels, L., Radiation of Uranium Solutions and Estimation of Uranium by Measurement of Radio-Activity, 345

———, Radio-Activity of Solutions of Uranium Salts: Method for the Estimation of Uranium, 231

Mitchell, L. C., *and* Tolman, L. M., Composition of Different Varieties of Red Pepper, 418

Miyake, K., Method for the Estimation of Galactan, 158

Moeser, L., *and* Frank, G., Estimation of Phosphoric Acid, 344

Moewes, C., Estimation of Skatol and Indol Together in Fæces, 30

Moles, E., Ethylene Dibromide as Cryoscopic Solvent, 32

Moser, L., *and* Seeling, F., Estimation of Available Oxygen in Per-Salts by Means of Titanium Trichloride, 79

Mosher, M., *and* Curtmann, L. J., Detection of Tin, 297

Mosscrop, T. D., *and* Auld, S. J. M., Measurement of Tryptic Protein Hydrolysis by Determination of the Tyrosine liberated, 153

Mossler, G., Detection of Aloes in Extracts of Drugs Containing Oxymethylanthraquinones, 321

Muggenthaler, H., *and* Eibner, A., Hexabromide Number of Fatty Oils and the Estimation of Rape Oil in Raw Linseed Oils, 158

Müller, E., Iodimetric Estimation of Persulphuric Acid, 293

————, *and* Fichter, F., Volumetric Method for the Estimation of Tin by Means of Potassium Bromate, 229

————, *and* Jacob, W., Action of Persulphate on Iodate, and Estimation of Periodate in Presence of Persulphate, 479

———, E. R. E., Reduction of Manganese Dioxide by Nitrous Acid, and the Use of this Reaction in the Estimation of Phosphorus in Iron and Steel without Separation of Silicon, 77

———, P. T., New Rapid Method for the Bacteriological Examination of Water and its Application for the Testing of Springs and Filter-Beds, 30

Mumford, E. M., Estimation of Phenol in Presence of Organic Matter, 336

Murdfield, R., *and* Kickton, A., Composition of Port Wine, 368

Murray, B. L., Determination of Small Amounts of Caffeine: A Comparison of Methods, 456

Nash, L. M., The Detection of Adulteration in Linseed Oil, 7

Natus, B., Direct Estimation of Elementary Nitrogen by Means of Commercial Calcium Carbide, 287

Nesbitt, C. T., Apparatus for Estimating Dust in Gas, 48

Neumann, O., Hop Analysis, 562

Nicloux, M., Apparatus for Extracting Carbon Monoxide from Blood, 592

————, Estimation and Detection of Small Quantities of Methyl Alcohol in Blood and Animal Tissues, 62

————, Simultaneous Estimation of Methyl Alcohol and Formaldehyde, 576

Nicolardot, P., Analysis of Paints and Especially Lithopones, 334

Nissenson, H., Rapid Methods for Estimation of Antimony, 339

Nockmann, E., Analysis of Pastry Containing Eggs, 417

Nola, E. D., *and* Malatesta, G., Detection of Gold and Platinum, 476

Noll, A., Microscopical Detection of Protoplasmalipoids, especially in Muscular Tissue, 423

Nolly, H. de, Rapid Determination of Total Carbon in Steel, Cast Iron, and Iron Alloys, 442

Normann, W., *and* Hugel, E., Analysis of Hardened Oils, 432

Noyes, W. A., *and* Others, Preliminary Report of the Committee on Coal Analysis of the American Society for Testing Materials and the American Chemical Society, 374

Nye, L. L., *and* Harding, E. P., Rapid Control Method for the Determination of Oil in Grains, 57

Nyman, M., Researches on the Gelatinising Temperatures of Starch Granules, 74

Obermayer, E., Estimation of Coumarin in Melilotus, 145

O'Brien, W. J., *and* Dennis, L. M., Determination of Phosphorus in Commercial Acetylene, 46

Ofner, R., Estimation of Higher Alcohols in Spirits, 564

Ogilvie, J. R., Determination of Sucrose in Cane Molasses by the Double Polarisation Method, Using Invertase and Acid as Hydrolysts, 281

————, Use of Sulphurous Acid in making the Acid Reading in the Double Polarisation Method of Determining Sucrose, 282

————, Eastick, J. J., *and* Lindfield, J. H., Estimation of Traces of Iron in Cane and Beet Sugar Factory and Refinery Products, 59

Öholm, L. W., New Method for Determining the Diffusion of Dissolved Substances, 350
Okuda, Y., Estimation of Creatine, Creatinine, and Mono-Amino Acids in certain Fishes, Mollusca, and Crustacea, 26
Oosthuizen, J. du P., *and* Shedd, O. M., Enzymes of the Tobacco Plant, 506
Owen, F., *and* Bradbury, W. A., Estimation of the Carbonates and Hydroxides of Potassium and Sodium when Together in Solution, 166

Page, H. J., *and* Plimmer, R. H. A., Investigation of Phytin, 279
Palitzsch, S., *and* Sörensen, S. P. L., "Salt Error" in Colorimetric Estimation of Hydrogen Ion Concentration of Sea-Water, 394
Paneth, F., *and* von Hevesy, G., Solubility of Lead Sulphide and Lead Chromate, 476
Parker, A., *and* Campbell, C., Preparation and Analysis of Methane, 430
———, C. E., New Separating Funnel, 234
———, E. G., Studies on Cellulose, 331
Parkes, A. E., A Simple Test for Differentiating between Cocoa Butter and "Green Butter," 203
————, Estimation of Chlorine in Rag Flock, 582
Parkin, G., *and* Harding, E. P., Rapid and Accurate Gravimetric Method for Determining Milk-Fat in Evaporated Milk and Milk Powders, 147
————————, Rapid and Accurate Method for Determining Fat in Ice Creams, Cereals, and Chocolate, 558
Partis, J., Estimation of *B. coli communis* in Water, 574
Partridge, W., The Bacterial Testing of Disinfectants: A Practical Criticism, 197
————, The Composition of Milk, 255
Paterson, W. H., Chemical Examination of Liquid Fuels, 212
Patusi, C., *and* Camilla, S., Detection and Estimation of Xanthin Bases in Cocoa, Tea, and Coffee, and their Preparations, 26
Pelligrini, A., *and* Marino, L., Volumetric Estimation of Hypophosphorous Acid and Hypophosphites, 342
Pelly, R. G., Examination of the Oils from *Manihot Ceara* and *Funtumia Elastica*, and a Comparison of their Properties with those of Linseed and *Hevea Oils*, 259
Pence, C. M., Permanganate [Alkaline] in the Estimation of Some Organic Compounds, 215
Pertusi, C., *and* Camilla, S., Analysis of Ferro-Silicon, 76
————————, Detection and Estimation of Xanthine Bases in Cocoa, Tea, Coffee, and their Preparations, 60
————, *and* Gastaldi, E., Detection of Hydrocyanic Acid, 332
Peterson, G. O., *and* Bailey, E. H. S., Hickory Nuts and Hickory Nut Oil, 498
Pfister, R., *and* Leuze, W., Table of Calculated Dry Substance of Milk by the Fleischmann Formula, 324
Pfützer, G., *and* Riesenfeld, E. H., Spectroscopic Detection of Magnesium, Calcium, Strontium, *and* Barium, 584
Pieraerts, J., Estimation of Starch in Commercial Products, 472
Piest, C., Estimation of the "Degree of Bleaching" [Chemical Condition] of Cotton, 73
Pilkington, B., *and* Tartar, H. V., Comparative Study of the Composition of Hops Grown in Different Parts of the World, 366
————————, Effect of Kiln-Drying at 140° F. on the Composition of the Hop, 20
Pinoff, E., *and* Gude, K., Estimation of Pentoses in Presence of Other Sugars by Means of the Spectroscope, 333

Platt, C. B., (and with), Clark, A. H., *and* Wheeler, R. V., Volatile Constituents of Coal. Part III., 511

Plimmer, R. H. A., Hydrolysis of Organic Phosphorous Compounds by Dilute Acid and Dilute Alkali, 278

——————, *and* Page, H. J., Investigation of Phytin, 279

Poetschke, P., Determination of Sodium Borate in Soap, 461

Pope, G. S., Sampling Coal Deliveries, 330

Popp, M., Estimation of Phosphoric Acid Soluble in Citric Acid in Thomas Slag, 526

Porritt, B. D., The Chemistry of Rubber, *Review*, 546

Porst, C. E. G., *and* Crown, H. A., Polarimetric Method for Determination of Starch in Paper, 380

Power, F. B., Tutin, F., *and* Rogerson, H., Constituents of Hops, 459

Pozzi-Escot, M. E., Contribution to the Study of the Colour Reactions of Chlorates, 339

——————, Estimation of Manganese in the Presence of Iron, 225

Pratt, D. S., Roselle, 60

Preisinger, J., *and* Artmann, P., Estimation of Phosphorus in Iron and Steel, 291

Pressey, H. F., *and* Lipman, C. B., Contribution to Methods of Determining Nitrogen in Humus, 163

Prianischnikow, D., Estimation of Absorbed Bases in Soils, 424

Pribram, E., Halle, W., *and* Loewentstein, E., Colour Reactions of Triketohydrindene Hydrate (Ninhydrin), 579

Pritz, W. B., Guillanden, A., *and* Withrow, J. R., Detection of Traces of Copper, 167

Prussia, L., *and* Grimaldi, C., Maumené Figure of Oil of Turpentine, 338

Rachel, F., Detection of Formaldehyde in Foods, 497

Rae, W. N., *and* Joseph, A. F., Notes on Alcoholometry, 508

Raikow, P. N., New Method for the Identification of Methyl Alcohol, 379

Rao, V., *and* Tollens, B., Estimation of Cellulose by Means of Nitric Acid, 509

Rappuzzi, E., *and* Finzi, C., Rapid Method of Estimating Lead Peroxide in Minium, 343

Rasmussen, H. T. B., Behaviour of Certain Sugars with Diphenylamine and Hydrochloric Acid, 435

Rather, J. B., Phytic Acid in Cottonseed Meal and Wheat Bran, 419

Rau, O., Estimation of Water Formed on Burning Substances Containing Nitrogen and Sulphur in a Calorimetric Bomb, 347

Rawlins, H. B. J., The Direct Volumetric Determination of Tin, 82

Redman, L. V., Weith, A. J., *and* Brock, F. P., Drying Rates of Raw Paint Oils, 468

——————————————, Effect of Temperature, Acid Concentration, and Time in the Bromination of Phenol for Quantitative Determinations, 335

Reeve, C. S., *and* Lewis, R. H., Application of the Dimethyl Sulphate Test for Determining Small Amounts of Petroleum or Asphalt Products in Tars, 277

Reichard, C., Reactions of Digitalis Glucosides, Digitonin, 265

——————, Reactions of Digitoxine, 457

Revis, C., The Composition of Milk, 255

————, The Detection and Estimation of Benzoic Acid in Milk and Cream, 557

————, *and* Bolton, E. R., A Simple Test for Differentiating between Cocoa Butter and "Green Butter," 201

————, *and* Burnett, H. R., The Proportionate Determination of Cocoanut and Palm-Kernel Oil in Mixtures, 255

Rheinberger, E., *and* Mai, C., Estimation of Water in Cheese, 146

Richardson, C., Characteristics and Differentiation of Native Bitumens and their Residuals, 371

Richardson, F. W., Combined Estimation of Boric Acid and Fat in Cream, 141
——————, The Bacterial Testing of Disinfectants: A Practical Criticism, 197
——————, *and* Walton, W. K., A Quick and Accurate Method for the Estimation of Boric Acid in Milk and Cream, 140
Richmond, H. D., Note on Samna, 252
——————, The Composition of Milk, 252
——, The Laboratory Book of Dairy Analysis, *Review*, 303
Combined Estimation of Boric Acid and Fat in Cream, 142
, The Bacterial Testing of Disinfectants: A Practical Criticism, 198
Richter, E., Estimation of Quinine by Titration with Picric Acid, 105
—————, New Technical Determination of Bleaching Quality of Sulphite Pulp, 72
Rideal, E. K., The Bacterial Testing of Disinfectants: A Practical Criticism, 199
—————, The Detection and Estimation of Benzoic Acid in Milk and Cream, 557
—————, *and* Evans, U. R., An Electrochemical Indicator for Oxidising Agents, 353
———, S., The Bacterial Testing of Disinfectants: A Practical Criticism, 198
————, The Detection of Adulteration in Linseed Oil, 7
————, An Electrochemical Indicator for Oxidising Agents, 363
and Acland, L. H. D., Examination of the Oils from *Manihot Ceara* and *Funtumia Elastica* and a Comparison of their Properties with those of Linseed and *Hevea* Oils, 259
Riesenfeld, E. H., *and* Pfützer, G., Spectroscopic Detection of Magnesium, Calcium, Strontium, and Barium, 584
Rivera-Maltes, —, Kohn-Abrest, E., *and* Rochas, —, Action of "Activated" Aluminium on Alkaloid Extracts and its Use in Toxicology, 268
Robertson, I. W., *and* Allen, I. C., Determination of Sulphur in Fuels, especially Petroleum Products, 38
Robin, L., Detection of Benzoic Acid in the Presence of Phenol and Salicylic Acid, 371
Rochas, —, Kohn-Abrest, E., *and* Rivera-Maltes, —, Action of "Activated" Aluminium on Alkaloid Extracts and its Use in Toxicology, 268
Rocques, X., Eaux-de-Vie: Eaux-de-Vie Naturelles et Industrielles, *Review*, 239
Rodano, G. A., *and* Armani, G., Estimation of Paraffin Wax and Ceresin in their Mixtures, 37
Rogée, H., *and* Fritsch, C., Estimation of Chlorine in Blood, 461
Rogerson, H., Power, F. B., *and* Tutin, F., Constituents of Hops, 459
Rohland, P., Estimation of Colloids in Effluents, 269
Rosam, A., Simple Method for Determining the Fermentative Activity of Milk and Various Feeding-Stuffs, and the Catalase Enzyme of Milk, 328
Rosenthal, E., Experiments on Antigen and Antibody Actions: Differentiation of Proteins (Kidney, Liver, and Cancer Proteins), 26
Rosenthaler, L., Detection of Saponin, 151
——————, *and* Abelmann, A., Mercuric Oxide as a Standard for Volumetric Analyses, 439
Ross, R., A Simple All-Glass Extraction Apparatus, 144
Rosset, H., Use of Miscibility Curves in the Examination of Spirits of Camphor, 204
Roth, E., *and* Kreis, H., Fractional Precipitation of Fatty Acid Mixtures (Detection of Rape Oil), 114
————————————, Solidified (Hydrogenised) Oils and the Detection of Arachidic Acid, 160
Rothenfusser, S., Detection of Cane-Sugar, 31
Rothmund, V., *and* Burgstaller, A., Estimation of Ozone and Hydrogen Peroxide, 344
Rousseaux, E., *and* Sirot, M., Estimation of Soluble Nitrogenous Substances as a Criterion of the Quality of Flour, 148
Rupp, E., *and* Mäder, H., Estimation of Hydroxylamine, 437

Ruttan, R. F., *and* Hardisty, R. H. M., New Reagent for the Detection of Traces of Blood, 422

Sacher, J. F., Method for the Analysis of Barium Sulphide, 75
———, Use of the Sense of Smell in Volumetric Estimations, 594
Sadtler, —, *and* Davis, —, Allen's Commercial Organic Analysis, Vol. VII., *Review*, 597
St. Rainer, L., Estimation of Platinum, 294
Saint-Sernin, A., Estimation of Calcium as Tungstate, 283
Salkowski, E., Precipitation of Purine Bases from Meat Extract and Urine by Means of Zinc Salts, 572
———, Precipitation of Uric Acid and Purin Bases by Zinc Salts, 424
Salvaterra, H., Comparative Investigation of Methods for the Estimation of Dyes, 113
Sammet, C. F., Quantitative Determination of Rosin in Paper, 516
———, O., Estimation of Acetone in Urine, 110
Sanarens, J., Composition of Rums, 500
Sand, H. J. S., *and* Trotman, S. R., Measurement of the Absorption of Oxygen by Sewage Effluents, 123
Sarin, E. J., Composition of Russian Honey, 149
Saul, J. E., Delicate Colour Reactions of Gold Salts, 54
Savage, W. G., Report to the Local Government Board on Bacterial Food-Poisoning and Food Infections, 535
Savini, G., *and* Silvestri, G., Estimation of the Quality of Flour, 55
Schaap, A., *and* de Graaff, W. C., Estimation of Proteins in Milk by Aldehyde Titration, 266
Schaefer, J. A., Determination of Lead Sulphate and the Ultimate Estimation of "Sublimed White Lead" in Rubber, 44
Schaffnit, D., Biological Considerations in Relation to Seed-Testing, 210
Schapira, A., *and* Wunder, M., Separation of Tungsten from Thorium, Lanthanum, Cerium, Erbium, Didymium, and Silica, 481
Schewket, O., Colour Reaction for Gallic Acid and Tannin, 467
Colour Reactions of Alkaline Earths with Oxygallol Derivatives, 473
———, New Colour Reactions of Di- and Tri-Phenols, 470
Schiller, W., *and* Kardos, M., Improved Apparatus for Extracting Finely Divided Solids with Volatile Solvents, 444
Schilling, H., Gravimetric Estimation of Zinc, 47
———, *and* Marcusson, J., Differentiation of Animal and Vegetable Fats, 458
Schinner, A., *and* Vanino, L., Interaction between Formaldehyde and Nitrous Acid, 67
———, New Hydrogen Table, 167
Schjerning, H., Nitrogenous Substances in Barley and Malt. III.: Transformation of the Proteins during Mashing, Boiling of Wort, and Fermentation, 496
Schloesing, T., Junr., Detection and Estimation of White Phosphorus in Phosphorus Sesquisulphide, 80
Schmidt, E., Detection of Gelatin in Textile Dressings, 332
———, W., Precipitation of Palladium by Means of α-nitroso-β-naphthol, 289
Schmiedel, R., Detection and Estimation of Methyl Alcohol, 509
Schmitz, W., *and* Tschirch, A., Method for the Estimation of the Nitrogenous Constituents of Raw Caoutchouc, 272
Schnabel, K., *and* Zänker, W., Detection of Free Sulphuric Acid in Cotton, 510
Schneeberger, P., *and* Gilpin, J. E., Fractionation of California Petroleum by Diffusion through Fuller's Earth, 469
Schneider, L., Estimation of Fluorine in Zinc Ores, 475
Schowalter, E., Colorimetric Estimation of Small Quantities of Manganese in Water, 477
Schröder, F., Detection of White Phosphorus in Match Compositions, 289

Schröder, F., Oil from the Seeds of *Ximenia Americana*, 147

Schulz, F., Volumetric Method for the Estimation of Sulphur in Illuminating Gas, 228

Schürmann, E., *and* Böttcher, W., Estimation of Arsenic in Pyrites, 117

Schütte, O., Rapid Volumetric Method for Estimation of Fatty Acids in Soaps, 432

Schutz, G., *and* Wein, L., Detection of Added Water in Milk by Means of the Refractometer Value of the Serum, 500

Schwartz, R., Use of the Immersion Refractometer in Examining American Beers made from Malt and Unmalted Cereals, 456

Schwarz, F., Detection of Natural Asphaltum and Petroleum Pitch in Residues from the Distillation of Coal-Tar, 157

———, *and* Marcusson, J., Examination of Steam Turbine Oils, 468

Schwinger, E., Melting-Point Apparatus for High Temperatures, 483

Scott, A. R., Analysis of Ferrotitanium, 474

Seeling F., *and* Moser, L., Estimation of Available Oxygen in Per-Salts by Means of Titanium Trichloride, 79

Seibert, F. M., *and* Burrell, G. A., Apparatus for Gas Analysis Laboratories at Coal Mines, 482

———, Apparatus for the Exact Analysis of Flue Gas, 351

Self, P. A. W., *and* Harrison, E. F., Analytical Constants of Extract of Male Fern, 429

Sendhoff, —, *and* Weinstein, —, Adulteration of Barley Meal, 264

Settim, L., *and* Maurantonio, L., Detection of Vegetable Oils in Margarine, 21

Settimj, L., Colour Reaction of Soya Bean Oil, 36

———, Detection of Mineral Oils in Oleins of Wool Fat, 35

Shedd, O. M., *and* Oosthuizen, J. du P., Enzymes of the Tobacco Plant, 506

Shipsey, K., *and* Werner, E. A., Purification of Acetone by Means of Sodium Iodide, 424

Shiro Tashiro, New Method and Apparatus for the Estimation of Extremely Small Quantities of Carbon Dioxide, 534

Shrewsbury, H. S., The Value of the Guaiacum Test for Bloodstains, 186

Sidenerand, C. F., *and* Skartvedt, P. M., Determination of Phosphorus in Vanadium Steel and Ferrovanadium, 587

Siegmund, I. W., Use of Hydrosulphite in Volumetric Analysis [of Dyestuffs], 32

Silberstein, S., *and* Bardach, B., Estimation of Glycerol in Sweet Raisin Wines, 24

Silvestri, G., *and* Cappelli, A., Detection of Nickel in Plated Articles, 45

———, *and* Savini, G., Estimation of the Quality of Flour, 55

Simmermacher, W., Estimation of Phosphoric Acid Soluble in Citric Acid in Thomas Slag, 171

Sinnatt, F. S., The Determination of Carbon Dioxide in Carbonates, 136

Sirot, M., *and* Rousseaux, E., Estimation of Soluble Nitrogenous Substances as a Criterion of the Quality of Flour, 148

Skartvedt, P. M., *and* Sidenerand, C. F., Determination of Phosphorus in Vanadium Steel and Ferrovanadium, 587

Skertchley, W. P., Determination of Moisture in Foods and Other Organic Substances, 177

Skinner, W. W., *and* Collins, W. D., Determination of Lithium, 168

Skopnik, A. von, *and* Marcusson, J., Distilled Wool Fat, 106

Smalley, F. N., Estimation of Total Fatty Acids in Cottonseed Foots, 33, 34

Smith, H. C., Chemistry for Dental Students, *Review*, 239

———, H. G., Apparatus for the Precipitation of Barium Sulphate, 350

———, H. L., The Determination of Tannin in Tea, 312

Smith, H. L., *and* Masters, H., Behaviour of Calcium and Magnesium Salts with Soap Solutions, and the Determination of Hardness of Water, 436
——, O. C., Modification of the Official Method of Determining Humus, 115
——, T. O., *and* James, C., Some Quantitative Separations of Neodymium, 343
Smorodinzew, J., Occurrence of Carnosine, Methylguanidine, and Carnitin, in Horseflesh, 508
Snapper, J., Chlorine Content of Blood and its Distribution between Serum and Blood-Corpuscles, 327
——, Neutral Red Paper as Indicator for Alkalimetry of Serum, 328
Snell, J. F., Analysis of Maple Products. I. Electrical Conductivity Test for the Purity of Maple Syrup, 499
Snelling, W. O., *and* Storm, C. G., Analysis of Black Powder and Dynamite, 514
——, Behaviour of Nitroglycerine when Heated, 115
Soper, G. A., Apparatus for the Determination of Dissolved Oxygen in Water, 396
——, *and* Parsons, P. B., Practical Field Method for the Determination of Dissolved Oxygen in Water, 397
Sörensen, S. P. L., *and* Palitzsch, S., "Salt Error" in Colorimetric Estimation of Hydrogen Ion Concentration of Sea-Water, 394
Southcombe, J. E., Chemistry of the Oil Industries, *Review*, 445
Soyka, W., *and* Meyer, H., Candelilla Wax, 514
Splittgerber, A., Total Solids of Milk, 21
Sprinkmeyer, H., *and* Diedrichs, A., Kapok Seeds and Kapok Oil, 467
Stansell, L. W., The Use of Glycerin-Jelly in Mounting Microscopical Objects, 407
Stansfield, J. F., *and* Elsden, A. V., Estimation of Small Quantities of Lead, 440
Stegmuller, P., *and* Fiehe, J., Composition of Honey, 265
Stephenson, H. H., Ceramic Chemistry, *Review*, 181
Stevens, —, An Electrochemical Indicator for Oxidising Agents, 362
——, Constant Temperature Apparatus, 311
——, The Preparation of Rubber for Analysis, 554
——, H. P., *and* Beadle, C., A Simple All-Glass Extraction Apparatus, 143
Stewart, R., *and* Greaves, J. E., Influence of Chlorine on Determination of Nitrates by Phenol Disulphonic Acid Method, 339
Stiepel, C., Detection of Marine Animal Oil (Thran) in Other Oils and Fats, 35
Stocklasa, J., Biochemical Examination of Soil, 462
Stoikoff, S., *and* Zlataroff, A., Composition of the Seeds of *Cicer Arietinum, L.* (Chick-Pea), 496
Storm, C. G., *and* Snelling, W. O., Analysis of Black Powder and Dynamite, 514
——, Behaviour of Nitroglycerine when Heated, 115
Streicher, L., *and* Matthes, H., Waxes and Resins of Kapok and Akon Fibres, 580
Strevens, J. F., *and* Dunstan, A. E., Viscosity of Lubricating Oils, 36
Strigel, A., *and* Dodt, J., Estimation of Potassium as Perchlorate in Potash Fertilisers, 124
Stritar, M. J., Estimation of Manganese in Soil, 343
Strohbach, E., Analysis of Cerium Compounds, 120
Stuckert, L., *and* Enderli, M., Bomb with Stirring Gear for the Measurement of Reaction-Velocity in a Heterogeneous System at High Pressures. New High-Pressure Reducing Valve, 590
Sugiura, K., *and* Kober, P. A., Micro-Chemical Method for the Estimation of α- and β-Amino-Acids and Certain Derivatives in Proteolysed Substances, Blood, and Urine, 567
Sundberg, T., Calculation of the Quantity of Cocoanut Oil Present in a Butter from the Polenske Value of the Mixture, 558
Sutcliffe, J. A. L., *and* Elsdon, G. D., Nitrates and Nitrites in Milk, 450
Sznajder, L., Rapid Estimation of Sulphur in Burned Pyrites, 529

Takahashi, T., Detection of Amyl Alcohol and Other Aliphatic Alcohols and their Esters, 574
Tamura, M., Oxidation of Fatty Acids and Unsaponifiable Matter during Process of Fat Estimation by the Kumagawa-Suto Method, 432
Tartar, H. V., *and* Pilkington, B., Comparative Study of the Composition of Hops Grown in Different Parts of the World, 366
————————, Effect of Kiln-Drying at 140° F., on the Composition of the Hop, 20
Tashiro, S., Method for Detecting the Vitality of Seeds, 370
Tassilly E., Estimation of Iron in Water, 174
Tellera, G., Detection of Paraffins in Lanolin, 458
Terni, A., Detection of Chromium, 437
Testoni, G., Estimation of Sucrose in the Presence of Other Sugars, 71
Thomas, A., *and* White, G. F., Studies on Fish Oils. III.: Properties of Fish and Vegetable Oil Mixtures, 70
———, V., *and* Boiry, F., Baobab Oil, 498
Thompson, A. R., Estimation of Sulphur and Chlorine in the Rice Plant, 572
————, G. W., Tests on the Opacity and Hiding Power of Pigments, 161
Thornton, W. J., jun., Estimation of Titanium in the Presence of Iron, 82, 229
Thüringer, V., *and* Wunder, M., Estimation of Palladium, and its Separation from Copper and Iron, 79
————————, Separation of Palladium from Gold, Platinum, Rhodium, and Iridium, 524
Tollens, B., *and* Rao, V., Estimation of Cellulose by Means of Nitric Acid, 509
Tolman, L. M., *and* Mitchell, L. C., Composition of Different Varieties of Red Pepper, 418
Treadwell, W. D., Electrolytic Separation of Copper from Tungsten and Molybdenum, 224
—————, *and* Guiterman, K. T., Separation of Cadmium from Zinc, 383
—————, Separation of Tin from Tungsten Electrically, 479
Tred, E. B., Investigations on the Quantitative Reduction of Methylene Blue by Milk Bacteria, and the Use of this Dye for Determining the Condition of Milk, 62
Trimen, S. H., Egyptian Butter and Samna, 242
Troccoli, A., *and* Verona-Rinati, G., Adulterated Peppercorns, 58
Trotman, S. R., *and* Sand, H. J. S., Measurement of the Absorption of Oxygen by Sewage Effluents, 123
Tschirch, A., *and* Schmitz, W., Method for the Estimation of the Nitrogenous Constituents of Raw Caoutchouc, 272
Tucker, F. H., *and* Cain, J. R., Determination of Phosphorus in Steels Containing Vanadium, 478
Tutin, F., Power, F. B., *and* Rogerson, H., Constituents of Hops, 459
Twining, R. H., *and* White, G. F., Viscosity of Butter Fat and its Substitutes, 458

Ueno, S., Fatty Acids of Kaya Oil, 458
Uhlinger, R. H., Lee, R. E., *and* Amon, F. O., Qualitative Analysis of the Zinc Group, 349
Utz, —, Estimation of Water in Cheese, 322

Vallery, M., Use of the Köttstorfer Value for the Estimation of Resins and Substitutes in Caoutchouc Articles, 337
Van den Bos, J. L. M., van der Horn, Cobaltinitrite Method for the Estimation of Potassium, 294

Van Eck, J. J., Use of Sodium Potassium Tartrate in the Estimation of Dissolved Oxygen in Water, 442

Vandevelde, A. J. J., Estimation of Phenols in Creolins, 325

Vanino, L., *and* Schinner, A., Interaction between Formaldehyde and Nitrous Acid, 67

——————————, New Hydrogen Table, 167

Veckenstedt, H., Ehrenberg, P., *and* Diebel, C., Estimation of Organic Matter in Clays, 374

Verona-Rinati, G., *and* Troccoli, A., Adulterated Peppercorns, 58

Vila, A., Detection of Adulteration by the Serum Precipitation Method, 152

Villavecchia, V., *and* Barboni, I., Estimation of Boric Acid in Charcuterie, 55

Ville, J., Methods of Sensitising Mylius's Reaction for the Detection of Cholalic Acid and Other Biliary Acids, 506

Vilmorin, P. L. de, *and* Levallois, F., Hydrolysis of Lævulosans and its Application for Analytical Purposes, 429

Voigt, K., Rapid Method of Estimating Zinc, 127

Vorisek, A., Cadmium Nitrate in Quantitative Analysis, 43

Vorländer, D., Berlin (Prussian) Blue Reaction (Detection of Ferrocyanides and Cyanides), 111

Votoček, E., Simple Reagent for the Detection of Mechanical Wood in Paper, 434

Vries, H. J. F. de, Estimation of Sodium Carbonate and Copper in Mixtures of Soda Ash and Blue Vitriol by Titration with Potassium Hydroxide, 340

Wagenaar, M., Behaviour of Salts of Cæsium and Rubidium towards Salts of Heavy Metals, 283

Wahl, R., Distinctions between Pepsin and Peptase, 507

Walbum, L. E., Red Cabbage Extract as Indicator for Measuring Hydrogen Ion Concentration, 214

Waldbott, S., Precipitation of Alkaloids by Lloyd's Reagent, 426

Walker, J., *and* Kay, S. A., Acidity and Alkalinity of Natural Waters, 40

Walton, W. K., *and* Richardson, F. W., A Quick and Accurate Method for the Estimation of Boric Acid in Milk and Cream, 140

Ward, H. L., Estimation of Lead, Nickel, and Zinc, by Precipitation as Oxalate and Titration with Permanganate, 122

——————, The Oxalate-Permanganate Method for Estimating Copper in Presence of Cadmium, Arsenic, Iron, or Lead, 122

Washburn, C. E. W., *and* Williams, G. Y., Precision Viscosimeter for Measurement of Relative Viscosity and the Relative Viscosities of Water at 0°, 18°, 25°, and 50° C., 399

Wassiljewa, P. J., *and* Chlopin, G. W., Method of Distinguishing Natural from Artificial Colouring Matters by Means of their Electrical Conductivity, 377

Waters, C. E., Evaporation Test for Mineral, Lubricating, and Transformer Oils, 333

——————, Simple Gasoline Gas Generator for Sulphur Determinations, 592

Wdowiszewski, H., Volumetric Estimation of Phosphorus in Steel by Macagno's Method, 526

Weaver, E. R., *and* McBride, R. S., Estimation of Sulphate in Ammonium Sulphate Solution, with Special Reference to the Testing of Illuminating Gas, 382

Wegelin, G., Application of Tantalum Electrodes to Electro-Analytical Estimation of Copper and Zinc, 481

Weich, A., *and* Devarda, A., Morres's "Alizarol" Test for Milk, 460

Wein, L., *and* Schutz, G., Detection of Added Water in Milk by Means of the Refractometer Value of the Serum, 500

Weinland, R. F., *and* Binder, K., New Delicate Reaction for Elementary Oxygen, 172

Weinstein, —, *and* Sendhoff, —, Adulteration of Barley Meal, 264

Weishut, F., Methoxyl Estimations with Hydriodic Acid in Presence of Phenol, 33

Weiss, J. M., Estimation of Phenol in Crude Carbolic Acid and Tar Oils, 162
——, M., Colorimetric Method for the Estimation of Histidine, 578
Weisser, F., Estimation of Cyanogen Compounds in Gas Liquor, 31
Weisweiller, G., *and* Bertrand, G., Volatile Constituents of Roasted Coffee, 417
Weith, A. J., Redman, L. V., *and* Brock, F. P., Drying Rates of Raw Paint Oils, 468
————————————, Effect of Temperature, Acid Concentration, and Time in the Bromination of Phenol for Quantitative Determinations, 335
Welker, W. H., *and* Marsh, H. L., Aluminium Hydroxide as a Protein Precipitating Reagent in the Estimation of Lactose in Milk, 418
————, *and* Marshall, J., Precipitation of Enzymes from their Solutions by Means of Moist Aluminium Hydroxide, 423
Wendler, O., Estimation of Fat in Cheese by the "Neusal" Method, 146
Werner, E. A., *and* Coade, M. E., Estimation of Nitrites by Means of Thiocarbamide, and the Interaction of Nitrous Acid and Thiocarbamide in Presence of Acids of Different Strength, 440
————, *and* Shipsey, K., Purification of Acetone by Means of Sodium Iodide, 424
Wesson, L. G., Direct Determination of Rubber, 337
West, T., Determination of Oxygen in Copper and Brass, 520
Wheeler, R. V., Volatile Constituents of Coal. Part IV. Relative Inflammabilities of Coal Dusts, 512
————, *and* Clark, A. H., and with Platt, C. B., Volatile Constituents of Coal. Part III., 511
Whitaker, A., Improvement in Gas Thermo-Regulators, 351
White, G. F., *and* Thomas, A., Studies on Fish Oils. III. Properties of Fish and Vegetable Oil Mixtures, 70
————, *and* Twining, R. H., Viscosity of Butter Fat and its Substitutes, 458
——, W., *and* Larsen, C., Dairy Technology, *Review*, 544
Wiechowski, W., *and* Bass, R., Estimation of Purine Bases in Blood, 107
Willcox, —, The Value of the Guaiacum Test for Bloodstains, 189
Williams, G. Y., *and* Washburn, C. E. W., Precision Viscosimeter for Measurement of Relative Viscosity and Relative Viscosities of Water at 0°, 18°, 25°, and 50° C., 339
——, H. G., Estimation of Hypochlorites, 224
Wilson, E. H., "Wood Oils" from China and Japan, 580
——, J. A., *and* McCandlish, D., Estimation of Alkali Sulphides, 393
——, S. R., *and* Barnebey, O. L., Application of Diphenylcarbazide ("Diphenylcarbohydrazide") as Indicator in Titration of Iron with Dichromate, 167
Windisch, R., Estimation of Copper by Means of Sodium Hypophosphite, 75, 520
Winkler, L. W., Detection and Colorimetric Estimation of Lead, Copper, and Zinc, in Supply-Water, 123
————, Detection of Arsenic by Means of Bettendorf's Reagent, 221
————, Detection of Potassium by Means of Tartaric Acid, 295
————, Estimation of Lithium in Mineral Waters, 531
————, ———— of Small Quantities of Hydrogen Sulphide in Natural Waters, 532
Winter, H. W., *and* Lander, G. D., Antipyrin in Toxicological Analysis, 97
Winton, A. L., Chloral Hydrate Test for Charlock, 366
————, *and* Berry, E. H., Chemical Composition of Authentic Vanilla Extracts, together with Analytical Methods and Limits of Constants, 206
Winzer, P., *and* Meimberg, —, Separation of Niobium and Tantalum by Means of Potassium Chloride in Hydrofluoric Acid Solution, 226

Wirth, F., Separation of Thorium from Monazite Sand by Means of Hypophosphorous Acid. Detection of Cerium, 443

Withrow, J. P., Pritz, W. B., *and* Guillanden, A., Detection of Traces of Copper, 167

Wolff, H., Analysis of Spirit Varnishes. Part I.: Investigation of the Solvent, 164

———, Estimation of Water in Bleached Shellac, 280

———, Examination of Turpentine Oil by the Methods of the German Pharmacopœia, 501

Woo, K. J., *and* Hart, E. B., Simple Method for the Determination of Carbon in Organic Materials, 465

Woodcock, R. C., *and* Kingzett, C. T., The Bacterial Testing of Disinfectants: A Practical Criticism, 190

Wren, H., Organometallic Compounds of Zinc and Magnesium (Chemical Monographs, No. I.), *Review*, 304

Wright, A. M., Chemical and Bacteriological Study of Fresh and Frozen New Zealand Lamb and Mutton, 27

———, C. H., Extension of Vieth's Table for Correcting the Specific Gravities of Milk, 460

Wunder, M., *and* Schapira, A., Separation of Tungsten from Thorium, Lanthanum, Cerium, Erbium, Didymium, and Silica, 481

———, *and* Thüringer, V., Estimation of Palladium, and its Separation from Copper and Iron, 79

———, Separation of Palladium from Gold, Platinum, Rhodium, and Iridium, 524

Wysor, H., Analysis of Metallurgical and Engineering Materials: A Systematic Arrangement of Laboratory Methods, *Review*, 134

Yoshikawa, J. Estimation of *d*-Lactic Acid in Body Fuids and Organs, 569

Zade, —, Application of Precipitin Reactions to Seed Identification, 210

Zänker, W., *and* Schnabel, K., Detection of Free Sulphuric Acid in Cotton, 510

Zeileis, A., *and* Langheld, K., Analysis of Mixtures of the Lower Fatty Acids, 273

Zerner, E., Detection of Formaldehyde with *p*-Nitrophenylhydrazine, 466

Zlataroff, A., *and* Stoikoff, S., Composition of the Seeds of *Cicer Arictinum, L.* (Chick-Pea), 496

Zöhren, F., Preparation of Petermann's Ammonium Citrate Solution, 338

Zsigmondy, R., Simple Apparatus for Ultrafiltration, 486

INDEX OF SUBJECTS.

ABSINTHE and Similar Liqueurs, Estimation of Essential Oils in, and in Alcoholic Solutions of Essential Oils, 418
Absorbed Bases in Soils, Estimation of, 424
Absorption Apparatus for Use in Nitrogen Estimations by Dumas' Method, 234
ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS, 19, 55, 105, 145, 204, 264, 321, 365, 414, 456, 496, 558
Acetates, Amyl, and Collodions, Analysis of, 365
Acetic and Formic Acids, Detection of, 275
Acetone in Urine, Estimation of, 110
———, Purification of, by Means of Sodium Iodide, 424
Acetylcelluloses. Analysis and Examination of Non-Inflammable Cellulose Esters, 577
Acetylene and Olefine Series, Hydrocarbons of the, Estimation of, in Mixtures of Gaseous Hydrocarbons, 158
————, Commercial, Phosphorus in, Determination of, 46
Acid and Invertase as Hydrolysts, Determination of Sucrose in Cane Molasses by the Double Polarisation Method, Using, 281
Arachidic, the Detection of, and Solidified (Hydrogenised) Oils, 160
Benzoic, in Cream and Milk, the Detection and Estimation of, 555
———, in Meats, Estimation of, 414
———, in the Presence of Phenol and Salicylic Acid, Detection of, 371
Boric, and Fat, in Cream, Combined Estimation of, 141
——, in Charcuterie, Estimation of, 55
——, in Milk and Cream, the Estimation of, a Quick and Accurate Method for, 140
-Butyrometric Method for the Estimation of Fat in Cheese and Milk Products, 145
———, Estimation of Fat in Cheese by, without the Use of Amyl Alcohol, 146
Carbolic, Crude, and Tar Oils, Phenol in, Estimation of, 162
Cholalic, and Other Biliary Acids, the Detection of, Method of Sensitising Mylius's Reaction for, 506
Citric, in Wines, Detection of, 55
Phosphoric Acid Soluble in, in Thomas Slag, Estimation of, 171, 526
—, the Estimation of, in the Presence of Certain Other Acids, 12
——, Coal-Tar Dyes, Certain, Mixtures of, Quantitative Separation of, 112
——, Dilute, and Dilute Alkali, Hydrolysis of Organic Phosphorus Compounds by, 278
——, Embelic, Microscopical Detection of, 559
——, Formic, as a Solvent, 513
Detection and Estimation of, 378
Free, and Base, in Aluminium Sulphate, Estimation of, 283

Acid, Free Sulphuric, in Cotton, Detection of, 510
——, Gallic, and Tannin, Colour Reaction for, 467
——, Hydriodic, in Presence of Phenol, Methoxyl Estimations with, 33
——, Hydrochloric, and Diphenylamine, Behaviour of Certain Sugars with, 435
——, Hydrocyanic, Detection of, 332
——, ————, in Plants, the Determination of, with Special Reference to Kaffir Corn, Colorimetric Method for, 569
——, Hypophosphorous, and Hypophosphites, Volumetric Estimation of, 342
——, ——————, Separation of Thorium from Monazite Sand by Means of Detection of Cerium, 443
——, Iodic, Method of Estimating Bromine in Halogen Salts, 166
——, *d*-Lactic, in Body Fluids and Organs, Estimation of, 569
——, Lactic, in Urine, Estimation of, 328, 370
——, Molybdic, Sensitive Reaction for, 43
——, Nitric, Colour Reactions of Oils with Certain Phenols in the Presence of, 433
——, ———, Estimation of Cellulose by Means of, 509
——, ——— (Nitrates), in Milk, Detection of, 563
——, ———, in Sulphuric Anhydride, Quantitative Detection of, 78
——, ———, Nitrous Acid in the Presence of, Detection of, 585
, Solutions Containing, Copper in, Electrolytic Determination of, 340
Nitrous, and Formaldehyde, Interaction between, 67
————, and Thiocarbamide, in Presence of Acids of Different Strength, the Interaction of, and Estimation of Nitrites by Means of Thiocarbamide, 440
————, in the Presence of Ferric Salts, Detection of, 289
————, ———————— Nitric Acid, Detection of, 585
————, Reduction of Manganese Dioxide by, and the Use of this Reaction in the Estimation of Phosphorus in Iron and Steel, without Separation of Silicon, 77
Persulphuric, Iodimetric Estimation of, 293
Phosphoric, Estimation of, 344
——————, Quantitative Volatilisation and Separation of, from the Metals of the Copper Group, and from Aluminium, Tin, and the Alkali Metals, 525
——————, Separation of, by Means of Stannic Acid, in Qualitative Analysis, 292
Soluble in Citric Acid, in Thomas Slag, Estimation of, 171, 526
Phytic, in Cottonseed Meal and Wheat Bran, 419
Picric, Estimation of Quinine by Titration with, 105
-, the Precipitation of Albumin by, the Conditions of, Studies of, 329
Salicylic, and Phenol, Benzoic Acid in the Presence of, Detection of, 371
——, in Fruit Juices, Detection and Estimation of, 420
Selenic, and Sodium Selenate, Preparation of, as Reagents for the Estimation of Bromine in Halogen Salts, 295
Application of, to the Estimation of Bromide in Presence of Chloride, 227
–, Stannic, in Qualitative Analysis, Separation of Phosphoric Acid by Means of, 292
–, Succinic, in Meat Extract and Fresh Meat, Occurrence of, 508
–, Sulphurous, and Sulphites, Titration of, in Presence of Thiosulphates, New Process for, 297
–, Sulphurous, Free, and Thiosulphate, Estimation of Polythionate in Presence of, 392
–, ——————, in Air, Estimation of, 127
–, ——————, Use of, in Making the Acid Reading in the Double Polarisation Method of Determining Sucrose, 282
–, Tartaric, Action of, on Tin in the Presence of Oxygen, 472

Acid, Tartaric, Detection of Potassium by Means of, 295
    the Determination of, 380
    —, Sulphates, Calcium, and Magnesium in Wine, Physico-Chemical Estimation of, 421
  Tungstic, Ferrotungsten, Metallic Tungsten Powder, and Tungsten Oxide, Determination of Phosphorus in, by Direct Solution, 290
    —, Quantitative Precipitation of, by Means of Aromatic Amines, 529
——, Uric, and Purin Bases, Precipitation of, by Zinc Salts, 424
——, ——, in Urine, Estimation of, 65
——, ——, ————, the Estimation of, Colorimetric Method for, 154
——, ——, ————, Titration of, after Silver Precipitation, 329
Acidity and Alkalinity of Natural Waters, 40
Acids, Acetic and Formic, Detection of, 275
——, α- and β-Amino, and Certain Derivatives in Proteolysed Substances, Blood, and Urine, Micro-Chemical Method for the Estimation of, 567
——, Amino-, in Blood, 567
——, Biliary, Other, and Cholalic Acid, the Detection of, Method of Sensitising Mylius's Reaction for, 506
——, Boric and Phosphoric, Titration of, 124
——, Fatty, and their Isomeric Esters, Quantitative Study of the Absorption of Ultra-Violet Rays by the, 113
    and Unsaponifiable Matter, Oxidation of, during Process of Fat Estimation by the Kumagawa-Suto Method, 432
  ——, in Soap, Estimation of, Rapid Volumetric Method for, 432
  ——, of Kaya Oil, 458
  ——, the Lower, Analysis of Mixtures of, 273
  ——, Volatile, Detection of, 274
, Formic and Acetic, Detection of, 275
  Malic and Tartaric, in the Same Solution, Determination of, 381
  Mono-amino, Creatine, and Creatinine, in Certain Fishes, Mollusca, and Crustacea, Estimation of, 26
  Oleic and Stearic, Mixtures of, Solidifying and Melting-Points of, 579
  Organic, Determination of Carboxyl Groups in, Improved Apparatus for, 299
  Palmitic and Stearic, Present in Lard, Mixed Glycerides of, 204
  Phosphoric and Boric, Titration of, 124
  Stearic and Oleic, Mixtures of, Solidifying and Melting-Points of, 579
    and Palmitic, Present in Lard, Mixed Glycerides of, 204
——, Tartaric and Malic, in the Same Solution, Determination of, 381
——, Total Fatty, in Cottonseed Foots, Estimation of, 33, 34
Adulterated Peppercorns, 58
Adulteration, Detection of, by the Serum Precipitation Method, 152
———— in Linseed Oil, the Detection of, 3, 104
———— of Barley Meal, 264
————— Cider Vinegar with Distilled Vinegar, Detection of, 565
Aëration, Quantitative Ammonia Distillation by. III., 580
Agitator, Mechanical, 590
Air, Mine, the Examination of, Apparatus for, 128
— of Rooms, etc., where the Metal is used, Estimation of Mercury in, 477
—, Sulphurous Acid in, Estimation of, 127
Akon and Kapok Fibres, Waxes and Resins of, 580
Albumin, Casein and Globulin in Milk: Nephelometric Determination of Proteins, 571
——— in Urine, Detection of, 573
———, the Precipitation of, by Picric Acid, Studies of the Conditions of, 329
Alcohol, Absolute, the Preparation of, Application of Young's Method for, 424

Alcohol, Amyl, and Other Aliphatic Alcohols and their Esters, Detection of, 574
———, Methyl, and Formaldehyde, Simultaneous Estimation of, 576
Detection and Estimation of, 509
--, in Spirits, Estimation of, 56
--, Small Quantities of, in Blood and Animal Tissues, Estimation and Detection of, 62
-, The Identification of, New Method for, 379
———, Water in, Method for Estimating. Equilibria in Systems Containing Alcohols, Water, and Potassium Fluoride, 425
Alcoholometry, Notes on, 508
Alcohols, Higher, in Spirits, Estimation of 564
———, ———, in Wine, Estimation of, 23
———, Methyl and Ethyl, in the Presence of Each Other, Estimation of, 330
———, ——————, Small Quantities of, in Aqueous Solutions, Estimation of, 575
Alcoholysis and the Composition of Cocoanut Oil, 8
Aliphatic Alcohols, Other, and Amyl Alcohol, and their Esters, Detection of, 574
"Alizarol" Test for Milk, Morres's, 460
Aldehyde Reactions, Some, Quantitative Study of, 154
——— Titration, Estimation of Proteins in Milk by, 266
Alkali, Dilute, and Dilute Acid, Hydrolysis of Organic Phosphorous Compounds by, 278
——— Metals, Aluminium, and Tin, Quantitative Volatilisation and Separation of Phosporic Acid from the Metals of the Copper Group, and from, 525
——— Sulphides, Estimation of, 393
Alkalimetry of Serum, Neutral Red Paper as Indicator for, 328
Alkaline Earths, Colour Reactions of, with Oxygallol Derivatives, 473
[Alkaline] Permanganate in the Estimation of Some Organic Compounds, 215
Alkalinity and Acidity of Natural Waters, 40
Alkalis, the Permeability of Cells by, the Estimation of, Indicator Method for, 327
Alkaloid Extracts, Action of "Activated" Aluminium on, and its Employment in Toxicology, 61, 268
Alkaloids, Microsublimation of, under Reduced Pressure, 426
———, Precipitation of, by Lloyd's Reagent, 426
Alloys, Aluminium, Analysis of, 220
———, Antimony in, Detection of, 42
———, Copper-Tin, Analysis of, 388
———, Iron, Steel, and Cast Iron, Total Carbon in, Rapid Determination of, 442
———, Lead, Analysis of, and Cathodic Estimation of Lead, 582
——— of Lead, Tin, Antimony, and Copper, Analysis of, 584
———, White Metal, Copper in, Determination of, 222
Almonds, Bitter, Oil of, and Benzaldehyde, Estimation of, 19
Aloes in Extracts of Drugs Containing Oxymethylanthraquinones, Detection of, 321
Aluminium, "Activated," Action of, on Alkaloid Extracts, and its Employment in Toxicology, 61, 268
——— Alloys, Analysis of, 220
———, Amalgamated, Use of, in the Estimation of Tannin, 516
——— and Chromium, Quantitative Separation of: Analysis of Chromite, 473
——————, Separation of, and Precipitation of Aluminium Hydroxide, 517
——— Hydroxide as a Protein Precipitating Reagent in the Estimation of Lactose in Milk, 418

Aluminium Hydroxide, Moist, Precipitation of Enzymes from their Solutions by Means of, 423
——————, Precipitation of, and Separation of Aluminium and Chromium, 517
———— Sulphate, Free Acid and Base in, Estimation of, 283
————, the Separation of Titanium from, Use of "Cupferron" in, 390
————, Tin, and the Alkali Metals, Quantitative Volatilisation and Separation of Phosphoric Acid from the Metals of the Copper Group, and from, 525
Amines, Aromatic, Quantitative Precipitation of Tungstic Acid by Means of, 529
Amino Acids, α- and β-, and Certain Derivatives in Proteolysed Substances, Blood and Urine, the Estimation of, Micro-Chemical Method for, 567
Amino Acids in Blood, 567
Ammonia Distillation, Quantitative, by Aëration. III., 580
————, Folin's Method for the Determination of, Application of, to Fertilisers, 381
————, Quantitative Estimation of Trimethylamine in Presence of, 436
Ammonium Citrate, Neutral Solutions of, Methods for the Preparation of, 116
—————— Solution, Petermann's, Preparation of, 338
———— Sulphate, Estimation of Lactose in Milk by Precipitation with, 418
——————, Influence of, on the Polarimetric Estimation of Lactose, 217
——————— Solution, Estimation of Sulphate in, with Special Reference to the Testing of Illuminating Gas, 382
———— Superphosphates, Water-Soluble Phosphates in, Estimation of, 392
Amyl Acetates and Collodions, Analysis of, 365
—— Alcohol, and Other Aliphatic Alcohols and their Esters, Detection of, 574
Analysis and Preparation of Methane, 430
——— by Combustion, Cerium Dioxide as Contact Substance in, 519
———, Electro-, Development of, 341
———, Electrochemical, Small Apparatus for, 179
———, Elementary, and Heat of Combustion, Use of Bomb Calorimeter for, 533
———, Gas, Apparatus for, 395
———, Mineralogical, of Soils, 125
———, Toxicological, Antipyrin in, 97
Analytical Chemistry, Present Position and Importance of, 83
Anglo-Egyptian Sudan, Oil Seeds from the, 323
Anhydride, Nitrous, Gasometric Estimation of Nitric Oxide and Oxygen, Depending on the Formation of, 391
————, Sulphuric, Nitric Acid in, Qualitative Detection of, 78
Aniline Dyestuffs [Magenta] in Foods, and Especially in Wines, the Detection of, New Reaction for, 322
Animal and Vegetable Fats, Differentiation of, 458
——— Fats, Certain, the Detection of, Polenske "Difference Value" for, and its Theoretical Basis, 204
——— Tissues and Blood, Small Quantities of Methyl Alcohol in, Estimation and Detection of, 62
—————— Urine, the Estimation of Mercury in, Method for, 153
Anniversary Dinner, 96
Annual Address of the President, 88
Antigen and Antibody Actions, Experiments on: Differentiation of Proteins (Kidney, Liver, and Cancer Proteins), 26
Antimony, Arsenic, or Bismuth, Ores Containing, Electrolytic Estimation of Copper in, 222

Antimony, Electrolytic Deposition of, 581
———, Estimation of, Rapid Methods for, 339
——— in Alloys, Detection of, 42
———, Lead, Tin, and Copper, Alloys of, Analysis of, 584
Antipyrin in Toxicological Analysis, 97
Antiseptic Agents, Influence of, on the Reichert-Meissl Value of Fats, 68
Apophorometer, 302
Apparatus, a Simple All-Glass Extraction, 143
———, Absorption, for Use in Nitrogen Estimations by Dumas' Method, 234
——— and Method for Determining Hydrogen Sulphide in Illnminating Gas, 396, 593
——— and Method, New, for the Estimation of Extremely Small Quantities of Carbon Dioxide, 534
——— and Methods Used in Petroleum Testing. Part II.: Viscometry, 397
———, Constant Temperature, 308
———, Dialysing, 394
———, Electrically Heated, for Measuring Rotatory Power at Constant Temperature, 300
APPARATUS, ETC., ABSTRACTS, 48, 84, 128, 178, 231, 299, 350, 394, 444, 482, 533, 590
Apparatus, Extraction, for Solids and Liquids, 351
———, ———, Simple, 49
——— for Determining the Density and Internal Friction of Gases, 591
——— for Estimating Dust in Gas, 48
——— for Extracting Carbon Monoxide from Blood, 592
Liquids Decomposed by Heat, 444
for Gas Analysis, 395, 594
-- Laboratories at Coal Mines, 482
-- for Recovery of Evaporated Organic Solvents, 398
-- for the Continuous Evaporation of Large Volumes of Liquid under Diminished Pressure, 591
-- for the Determination of Dissolved Oxygen in Water, 396
— for the Exact Analysis of Flue Gas, 351
-- for the Examination of Mine Air, 128
— for the Precipitation of Barium Sulphate, 350
— for Use in the Estimation of Starch by Ewers' Process, 445
—, Hoffmann's, Estimation of Water in Vegetables by, 177
—, Improved, for Determination of Carboxyl Groups in Organic Acids, 299
for Extracting Finely-Divided Solids with Volatile Solvents, 444
--, for Testing the Jelly-Strength of Glues, 231
Melting-Point, for High Temperatures, 483
New, for Quantitative Dialysis, 232
Portable Pettersson-Palmqvist, 178
Simple, for Ultrafiltration, 486
Skita's Hydrogenising, Simple Modification of, 484
Small, for Electrochemical Analysis, 179
Apparatus, Specific Heat, New Designs for, 484
Aqueous Solutions, Small Quantities of Methyl and Ethyl Alcohols in, Estimation of, 575
(Arachis) Earthnut Oil, in Olive Oil, Detection of, by the Franz-Adler Method, 58
Arachis Oil, Composition of, 514
Arachidic Acid, the Detection of, and Solidified (Hydrogenised) Oils, 160
Aromatic Amines, Quantitative Precipitation of Tungstic Acid by Means of, 529
Arsenic and Tungsten, Separation of, 117
———, Ante-Mortem Administered, Distribution of, in the Human Body, 268

Arsenic, Antimony, or Bismuth, Ores Containing, Electrolytic Estimation of Copper in, 222

———, Cadmium, Iron, or Lead, Copper in the Presence of, the Oxalate-Permanganate Method for Estimating, 122

———, Detection of, by Means of Bettendorf's Reagent, 221

——— in Blood and Urine, Estimation of, 106

——— in Pyrites, Estimation of, 117

——— in Soils, the Estimation of, Factors Influencing, 221

Artificial Asphaltum, Natural in Presence of, Quantitative Estimation of, 156

——— Rum, Method of Distinguishing Jamaica Rum from, 368

Asbestos Mixtures, Magnesium Carbonate, etc., Magnesium Oxide in, Calorimetric Detection and Estimation of, 76

Ash Estimations, Calorimetric, 582

—, Soda, and Blue Vitriol, Mixtures of, Estimation of Sodium Carbonate and Copper in by Titration with Potassium Hydroxide, 340

Ashes, Plant, Fertilisers, and Soil Extracts, Potassium in, Estimation of, 47

Asphalt or Petroleum Products in Tars, Small Amounts of, Application of the Dimethyl Sulphate Test for Determining, 277

Asphaltum, Natural, and Petroleum Pitch, in Residues from the Distillation of Coal-Tar, Detection of, 157

Aspirin, Assay and Estimation of, 414

Atomic Weights, International, 1

Autenreith-Koenigsberger's Colorimeter, Colorimetric Methods for Water Analysis by the Use of, 179

Automatic Pipette, 534

Babcock Test, Modified, for Fat in Sweetened Ice Cream, 323

B. Coli Communis in Water, Estimation of, 574

Bacteria, Heat Evolved by, Micro-Calorimeter for Measuring, 421

———, Milk, Investigations on the Quantitative Reduction of Methylene Blue by, and the Use of this Dye for Determining the Condition of Milk, 62

Bacterial Food-Poisoning and Food Infections, Report to the Local Government Board on, 535

——— Testing of Disinfectants: A Practical Criticism, 190

Bacteriological Examination of Water, New Rapid Method for, and its Application for the Testing of Springs and Filter-Beds, 30

——— Methods of Water Analysis, with Special Reference to the Use of the Berkefeld Filter, 574

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC., ABSTRACTS, 26, 61, 106, 152, 208, 268, 327, 369, 421, 461, 504, 567

Balsam, Peru, Analysis of, 150

———, Reaction of, 58

I. Bang's Method of Sugar Estimation, 435

Baobab Oil, 498

Barium as Sulphate, Purification of the Precipitate Obtained in Estimating, 339

———, Magnesium, Calcium, and Strontium, Spectroscopic Detection of, 584

——— Sulphate, Natural and Artificial, Differentiation of, 517

———, the Precipitation of, Apparatus for, 350

——— Sulphide, the Analysis of, Method for, 75

Barley and Malt, Nitrogenous Substances in. III.: Transformation of the Proteins during Mashing, Boiling of Wort, and Fermentation, 496

——— Meal, Adulteration of, 264

Baryta Water, Action of, on Certain Sorts of Glass, 118

Base and Free Acid in Aluminium Sulphate, Estimation of, 283

Bases, Absorbed, in Soils, Estimation of, 424
  Purin, and Uric Acid, Precipitation of, by Zinc Salts, 424
  Purine, in Blood, Estimation of, 107
  ———, Precipitation of, from Meat Extract and Urine by Means of Zinc Salts, 572
  Pyridine, Directions for Testing, 471
  Xanthin, in Cocoa, Tea, and Coffee, and their Preparations, Detection and Estimation of, 26, 60
Basicity and Reaction of Soil, the Estimation of, Significance of, 462
Beers, American, Made from Malt and Unmalted Cereals, Use of the Immersion Refractometer in Examining, 456
Beet and Cane Sugar Factory and Refinery Products, Traces of Iron in, Estimation of, 59
Benzaldehyde and Oil of Bitter Almonds, Estimation of, 19
α-Benzildioxime, the Detection and Estimation of Nickel by Means of, 316
Benzoic Acid in Meats, Estimation of, 414
——————— Milk and Cream, the Detection and Estimation of, 555
——————— the Presence of Phenol and Salicylic Acid, Detection of, 371
Berkefeld Filter, the Use of, Bacteriological Methods of Water Analysis, with Special Reference to, 574
Berlin (Prussian) Blue Reaction (Detection of Ferrocyanides and Cyanides), 111
Bettendorf's Reagent, Detection of Arsenic by Means of, 221
Bichromate, Potassium, in Milk, Detection of, 563
Bile, Human, the Analysis of, 208
Biliary Acids, Other, and Cholalic Acid, the Detection of, Method of Sensitising Mylius's Reaction for, 506
Biochemical Examination of Soil, 462
Biological Considerations in Relation to Seed-Testing, 210
Bismuth, Arsenic, or Antimony, Ores Containing, Electrolytic Estimation of Copper in, 222
————, or Copper Compounds, and Formaldehyde, Interaction between, in the Presence of Alkali, 66
Bitter Almonds, Oil of, and Benzaldehyde, Estimation of, 19
Bitumens, Native, and their Residuals, Characteristics and Differentiation of, 371
Black Mustard Powder, 500
——— Powder and Dynamite, Analysis of, 514
Bleached Shellac, Water in, Estimation of, 280
Bleaches, Chlorine, Titration of, 340
"Bleaching, Degree of" [Chemical Condition] of Cotton, Estimation of, 73
Bleaching Quality of Sulphite Pulp, New Technical, Determination of, 72
Blood, Amino-Acids in, 567
——— and Animal Tissues, Small Quanties of Methyl Alcohol in, Estimation and Detection of, 62
Blood and Urine, Arsenic in, Estimation of, 106
  ———, Chlorides in, Estimation of, 270
———, Apparatus for Extracting Carbon Monoxide from, 592
———, Chlorine in, Estimation of, 461
——— -Corpuscles and Serum, Chlorine Content of Blood and its Distribution between, 327
———, Detection of, 568
——— of Frog and Tortoise, the Estimation of Sugar in the, Sources of Error in, 463
———, Proteolysed Substances, and Urine, the Estimation of α- and β- Amino Acids and Certain Derivatives in, Micro-Chemical Method for, 567

Blood, Purine Bases in, Estimation of, 107
——, Residual Reduction of, 327
—— -Serum, Lipoids in, Estimation of, 28
—— -Stains, the Value of the Guaiacum Test for, 186
——, Traces of, the Detection of, New Reagent for, 422
Blown Oils, Rosin in, Estimation of, 469
Body Fluids and Organs, δ-Lactic Acid in, Estimation of, 569
——, the Human, Distribution of Ante-Mortem Administered Arsenic in, 268
(Bohemian) Melnik Wine, 326
Boiling, Effect of, on the Physico-Chemical Behaviour of Human Milk, Cow's Milk, and Buttermilk, 205
——, of Wort, Mashing, and Fermentation, Transformation of the Proteins during (III.), Nitrogenous Substances in Barley and Malt, 496
Bomb Calorimeter, Use of, for Elementary Analysis and Heat of Combustion, 533
——, Calorimetric, Water Formed on Burning Substances Containing Nitrogen or Sulphur in, Estimation of, 347
—— with Stirring Gear for the Measurement of Reaction-Velocity in a Heterogeneous System at High Pressures. New High-Pressure Reducing Valve, 590

BOOKS, REVIEWS OF:
- A Handbook of Sugar Analysis: A Practical and Descriptive Treatise for Use in Research, Technical, and Control Laboratories, 235
- Alcool Méthylique, Vinaigres, 352
- Allen's Commercial Organic Analysis, Vol. V., 85
- ————, Vol. VII., 597
- An Introduction to the Physics and Chemistry of Colloids, 352
- ———— to the Study of Fuel, 488
- Analysis of Metallurgical and Engineering Materials: A Systematic Arrangement of Laboratory Methods, 134
- Beurres et Graisses Animales, 402
- Ceramic Chemistry, 181
- Chemistry for Dental Students, 239
- ———— of the Oil Industries, 445
- Chloride of Lime in Sanitation, 405
- Dairy Technology, 544
- Digitalis Assay, 237
- Eaux-de-Vie: Eaux-de-Vie Naturelles et Industrielles, 239
- Eaux Douces et Eaux Minérales, 51
- Farines, Fécules et Amidons, 545
- Fatty Foods, 50
- Food Inspection and Analysis, 490
- General Chemistry of the Enzymes, 84
- Industrial and Manufacturing Chemistry: Organic, 399
- Introduction to the Rarer Elements, 240
- Liquid Air, Oxygen, Nitrogen, 600
- Matériaux de Gros-Ouvre, 181
- Methods in Chemical Analysis Originated or Developed in the Kent Chemical Laboratory of Yale University, 489
- Notes on Chemical Research, 180
- Organometallic Compounds of Zinc and Magnesium (Chemical Monographs, No. I.), 304
- Tables Annuelles de Constantes et Données Numériques, 548
- The Application of Physico-Chemical Theory, 599
- The Chemistry of Rubber, 546

BOOKS, REVIEWS OF—*continued*
The Laboratory Book of Dairy Analysis, 303
The National Physical Laboratory. Collected Researches, 542
The Qualitative Analysis of Medicinal Preparations, 403
The Volatile Oils, 547
Theorien der Organischen Chemie, 601
Borate, Sodium, in Soap, Determination of, 461
Boric Acid and Fat in Cream, Combined Estimation of, 141
——— in Charcuterie, Estimation of, 55
——— in Milk and Cream, the Estimation of, a Quick and Accurate Method for, 140
——— and Phosphoric Acids, Titration of, 124
Bran, Wheat, and Cottonseed Meal, Phytic Acid in, 419
Brass, Analysis of, 436
——— and Copper, Oxygen in, Determination of, 520
Brisance, Estimation of, and of Velocity of Detonation, 464
Bromide in Presence of Chloride, the Estimation of, Application of Selenic Acid to, 227
Bromides and Chlorides in Organic Liquids, Determination of, 211
Bromination of Phenol for Quantitative Determinations, Effect of Temperature, Acid Concentration, and Time in, 335
Bromine Absorption of Wines, Estimation of the, 266
——— and Chlorine, New Reagent for, 119
——— and Iodine in Mineral Waters, etc., the Estimation of Hydrolytic Action a Source of Error in, 532
———, Characteristic Reaction of, 518
——— Free, Characteristic Reaction of, Oxidation with, Identification of Small Amounts of Dyestuffs by, 376
———, Traces of, Estimation of, 119
——— in Halogen Salts, Iodic Acid Method of Estimating, 166
———, the Estimation of, Preparation of Selenic Acid and Sodium Selenate as Reagents for, 295
——— Titration with, Experiments on the Direct Estimation of Caoutchouc by, 272
"Burette, Sulphuretted Hydrogen," Valuation of Hydrated Oxide of Iron by Means of the, 522
Butter, and Samna, Egyptian, 242
Butter, Cocoa, and "Green Butter," a Simple Test for Differentiating between, 201
——— Fat and its Substitutes, Viscosity of, 458
———, the Quantity of Cocoanut Oil Present in a, Calculation of, from the Polenske Value of the Mixture, 558
Buttermilk, Human Milk, and Cow's Milk, the Physico-Chemical Behaviour of, Effect of Boiling on, 205

Cabbage Extract, Red, as Indicator for Measuring Hydrogen Ion Concentration, 214
Cadmium, Arsenic, Iron, or Lead, Copper in Presence of, the Oxalate-Permanganate Method for Estimating, 122
——— Nitrate in Quantitative Analysis, 43
———, Separation of, from Zinc, 383
Cæsium and Rubidium, Salts of, Behaviour of, towards Salts of Heavy Metals, 283
Caffeine, Small Amounts of, Determination of: A Comparison of Methods, 456
Calcium and Magnesium Salts, Behaviour of, with Soap Solutions, and the Determination of Hardness of Water, 436

Calcium as Tungstate, Estimation of, 283
——— Carbide, Commercial, Direct Estimation of Elementary Nitrogen by Means of, 287
——— Citrate, Commercial, Analysis of, 65
———, Magnesium, Strontium, and Barium, Spectroscopic Detection of, 584
———, Sulphates, Tartaric Acid, and Magnesium in Wine, Physico-Chemical Estimation of, 421
———, Volumetric Estimation of, 518
California Petroleum, Fractionation of, by Diffusion through Fuller's Earth, 469
Calorimeter, Bomb, Use of, for Elementary Analysis and Heat of Combustion, 533
Calorimetric Ash Estimations, 582
———— Bomb, Estimation of, Water Formed on Burning Substances Containing Nitrogen or Sulphur in, 347
Camphor, Spirits of, the Estimation of, Use of Miscibility Curves in, 204
Canada, Coals of, 577
Candelilla Wax, 514
Cane and Beet Sugar Factory Refinery Products, Traces of Iron in, Estimation of, 59
—— Molasses, Sucrose in, Determination of, by the Double Polarisation Method, Using Invertase and Acid as Hydrolysts, 281
—— Products, Methods of Analysis of, Investigations on, 415
—— Sugar, Detection of, 31
Canned Foods, the Estimation of Tin in, Iodine Titration Method for the Estimation of Tin, especially in Connection with, 23
Caoutchouc Articles, the Estimation of Resins and Substitutes in, Use of the Köttstorfer Value for, 337
———— in Crude Rubber, Estimation of, 511
———— in Rubber Wares, Particularly Insulated Wires, Estimation of, 111
————, Raw, the Nitrogenous Constituents of, Method for the Estimation of, 272
————, Resins, Studies on, 465
————, the Direct Estimation of, by Titration with Bromine, Experiments on, 272
Carbohydrates, Especially in Plant Extracts, Methods of Estimation of, 504
Carbolic Acid, Crude, and Tar Oils, Phenol in, Estimation of, 162
Carbon Dioxide, Estimation of, 385, 437
—————, Extremely Small Quantities of, the Estimation of New Method and Apparatus for, 534
—————, Gravimetric Estimation of, 386
————— in Carbonates, the Determination of, 136
Carbon, Estimation of, by Combustion with Moist Oxygen, 271
———, in Iron and Steel, Rapid Method for Estimating, 384
——— in Organic Materials, the Determination of, Simple Method for, 465
——— Monoxide, Apparatus for Extracting, from Blood, 592
——————, Small Quantities of, Estimation of, 75
——— Tetriodide in the Presence of Iodoform, Estimation of, 373
———, Total, in Steel and Ferro-Alloys, Estimation of, by Combustion in Oxygen under Pressure, 285
———, Total, in Steel, Cast Iron, and Iron Alloys, Rapid Determination of, 442
Carbonate, Magnesium, Asbestos Mixtures, etc., Magnesium Oxide in, Calorimetric Detection and Estimation of, 76
Carbonates and Hydroxides of Potassium and Sodium when Together in Solution, Estimation of, 166
————, Carbon Dioxide in, the Determination of, 136
———— in Soils, Some Qualitative and Quantitative Methods for, Comparison of, 163
Carboxyl Groups in Organic Acids, Determination of, Improved Apparatus for, 299

Carnitin, Carnosine, and Methylguanidine, in Horseflesh, Occurrence of, 508
Carnosine, Methylguanidine, and Carnitin, in Horseflesh, Occurrence of, 508
Casein and Lactose in Milk, Estimation of, 21
———, Globulin, and Albumin in Milk: Nephelometric Determination of Proteins, 571
Cast Iron, Steel, and Iron Alloys, Total Carbon in, Rapid Determination of, 442
Castings and Steels, Manganese in, Estimation of, by the Method of G. von Knorre, 286
Catalase Enzyme of Milk, and Simple Method for Determining the Fermentative Activity of Milk and Various Feeding-Stuffs, 328
Cathodic Estimation of Lead and Analysis of Lead Alloys, 582
Cell Elements in Milk, the Distribution of, Influence of Centrifugal Action on, 108
Cells, the Permeability of, by Alkalis, Indicator Method for the Estimation of, 327
Cellulose and Crude Fibre, Estimation of, 415
——— Esters, Non-Inflammable, Analysis and Examination of Acetylcelluloses, 577
———, Estimation of, by means of Nitric Acid, 509
———, Studies on, 331
Cement, Portland, Examination of, 437
Cereals, Ice Creams, and Chocolate, Fat in, Rapid and Accurate Method for Determining, 558
Ceresin and Paraffin Wax in their Mixtures, Estimation of, 37
Cerium Compounds, Analysis of, 120
———, Detection of. Separation of Thorium from Monazite Sand by Means of Hypophosphorous Acid, 443
——— Dioxide as Contact Substance in Analysis by Combustion, 519
———, Thorium, Lanthanum, Erbium, Didymium, and Silica, Separation of Tungsten from, 481
Characteristic Reaction of Bromine, 118, 518
Charcuterie, Boric Acid in, Estimation of, 55
Charlock, Chloral Hydrate Test for, 366
Cheese and Milk Products, the Estimation of Fat in, Acid-Butyrometric Method for, 145
———, Fat in, Estimation of, 417
———, ———, ———, by the Acid-Butyrometric Method without the Use of Amyl Alcohol, 146
———, by the "Neusal" Method, 146
———, Water in, Estimation of, 146, 322
"Cheyi" Seed, 323
(Chick-Pea) *Cicer Arietinum*, L., the Seeds of, Composition of, 496
Chicory and Coffee, Note on, 454
China and Japan, "Wood Oils" from, 580
Chloral Hydrate Test for Charlock, 366
Chlorate Solutions, the Rendering Active of, and Osmium Tetroxide as an Oxygen-Carrier, 78
Chlorates and Stannous Chloride, Determination of, by Means of Methylene Blue, 126
———, the Colour Reactions of, Contribution to the Study of, 339
Chloride, the Estimation of Bromide in Presence of, Application of Selenic Acid to, 227
Chlorides and Bromides in Organic Liquids, Determination of, 211
——— in Urine and Blood, Estimation of, 270

Chlorides in Wine, Estimation of, 502
Chlorine and Bromine, New Reagent for, 119
——— and Sulphur in the Rice Plant, Estimation of, 572
——— Bleaches, Titration of, 340
——— Content of Blood and its Distribution between Serum and Blood-Corpuscles, 327
——— in Blood, Estimation of, 461
——— in Rag Flock, Estimation of, 582
——————, the Estimation of, Note on, 409
———, Influence of, on Determination of Nitrates by Phenol Disulphonic Acid Method, 339
Chloroform, Examination of, 497
Chocolate and Cocoa, Copper in, 145
————, Ice Creams, and Cereals, Fat in, Rapid and Accurate Method for Determining, 558
Cholalic Acid and Other Biliary Acids, the Detection of, Method of Sensitising Mylius's Reaction for, 506
Cholesterol Bodies in Presence of Each Other, Estimation of, 466
————, the Oxydation Products of, Study of, 209
Chromate, Lead, and Lead Sulphide, Solubility of, 476
Chromic Oxide, Free and Combined, Iodimetric Estimation of, 386
Chromium and Aluminium, Quantitative Separation of: Analysis of Chromite, 473
——————————, Separation of, and Precipitation of Aluminium Hydroxide, 517
———— and Iron, Quantitative Separation of, 387
———— and Manganese, New Separation of, 474
——————————, Separation of, 474
—, Small Quantities of, in Minerals and Rocks, Estimation of, 226
———— and Vanadium in Steels, Determination of, 81
————, Detection of, 437
———— in Presence of Iron, Volumetric Estimation of, 387
———— in Steel, Determination of, Colorimetric Method for, 285
————, Vanadium, and Iron, in Presence of One Another, the Volumetric Estimation of, a New Method for, 99
Cider Vinegar, Adulteration of, with Distilled Vinegar, Detection of, 565
Citric Acid in Wines, Detection of, 55
—————, Phosphoric Acid Soluble in, in Thomas Slag, Estimation of, 171, 526
—————, the Estimation of, in the Presence of Certain Other Acids, 12
———, Tartaric, Lactic, and Malic Acids in Musts, Wines, etc., Estimation of, 504
Clays, Organic Matter in, Estimation of, 374
Coal Analysis, Accuracy and Limitations of, 211
——————, the Committee on, of the American Society for Testing Materials, and the American Chemical Society, Preliminary Report of, 374
—— Deliveries, Sampling, 330
—— Dusts, Relative Inflammabilities of. Part IV. Volatile Constituents of Coal, 512
—— Mines, Gas Analysis Laboratories at, Apparatus for, 482
——-Tar Dyes, Certain Acid, Quantitative Separation of Mixtures of, 112
————, the Distillation of, Residues from, Detection of Natural Asphaltum and Petroleum Pitch in, 157
——, Volatile Constituents of. Part III., 511
——————. Part IV. Relative Inflammabilities of Coal Dusts, 512
Coals of Canada, 577

Cobalt and Uranium in Steel, Estimation of, 527
Cobalitnitrite Method, Estimation of Potassium by, 81
—————————— for the Estimation of Potassium, 294
Cocoa and Chocolate, Copper in, 145
——— Butter and "Green Butter," a Simple Test for Differentiating between, 201
———, Essential Oil of, 20
———, Tea, and Coffee, and their Preparations, Xanthin Bases in, Detection and Estimation of, 26, 60
Cocoanut and Palm-Kernel Oil in Mixtures, the Proportionate Determination of, 255
———— Oil, the Composition of, and Alcoholysis, 8
——————, the Quantity of, Present in a Butter, Calculation of, from the Polenske Value of the Mixture, 558
Coffee and Chicory, Note on, 454
———, Cocoa, and Tea, and their Preparations, Xanthin Bases in, Detection and Estimation of, 26, 60
———, Roasted, Volatile Constituents of, 417
Cohune Nuts, Oil of, 433
"Cold Test" of Oil, Experiments on the, 34
Collodions and Amyl Acetates, Analysis of, 365
Colloids in Effluents, Estimation of, 262
————, Influence of, on the Electrolytic Separation of Lead, 76
Colophony in Shellac, Small Amounts of, the Detection of, and Shellac Analysis, 38
Colorimeter, Autenreith-Koenigsberger's, Colorimetric Methods for Water Analysis by the Use of, 179
Colorimetric Estimation and Detection of Lead, Copper, and Zinc, in Supply-Water, 123
———————————— of Hydrogen Ion Concentration of Sea-Water, "Salt Error" in, 394
— of Small Quantities of Manganese in Water, 225, 477
Method for Determination of Chromium in Steel, 285
———— for the Determination of Hydrocyanic Acid in Plants, with Special Reference to Kaffir Corn, 569
———— for the Estimation of Histidine, 578
of Uric Acid in Urine, 154
for Titanium in Iron and Steel, 588
Colostrum and Colostrum Fat, Composition of, 107
Colour Method, the Hydrogen Peroxide, Estimation of Vanadium in Steel by, 589
——— Reaction for Gallic Acid and Tannin, 467
——————— of Hypochlorites with Methylaniline and Ethylaniline, 476
——————— of Proteins, 380
——————— of Soya Bean Oil, 36
of Wine, 24
—— Reactions, Delicate, of Gold Salts, 54
-, New, of Di- and Tri-Phenols, 470
———— of Alkaline Earths with Oxygallol Derivatives, 473
———— of Certain Indole Derivatives and their Significance with regard to the Glyoxylic Reaction, 276
— of Chlorates, the Study of, Contribution to, 339
—— of Hardened Whale Oil, 433
—— of Oils with Certain Phenols in the Presence of Nitric Acid, 433
—— of Triketohydrindene Hydrate (Ninhydrin), 579
Coloured Fluids, International Standards for, and a Suggested Plan for such Standardisation, 394

Colouring Matters, Method of Distinguishing Natural from Artificial, by Means of their Electrical Conductivity, 377
Colours, Food, the Permitted, Quantitative Separation and Determination of Subsidiary Dyes in, 375
Combustion, Analysis by, Cerium Dioxide as Contact Substance in, 519
——— Method, the Liebig, Modification of, 301
Concentration of Hydroxyl Ions, the Determination of, New Method for, 522
Conductivity, Test (I.) Electrical, for the Purity of Maple Syrup. Analysis of Maple Products, 499
——— Water, Preparation of, 346
Confectionery, Saffron in, Detection of, 206
Connecticut Agricultural Experiment Station, Report of, for the year 1912, 541
Constant Temperature Apparatus, 308
———, Measuring Rotatory Power at, Electrically Heated Apparatus for, 300
Contact Substance in Analysis by Combustion, Cerium Dioxide as, 519
Copper and Brass, Oxygen in, Determination of, 520
——— and Iron, Estimation of Palladium, and its Separation from, 79
——— and Sodium Carbonate in Mixtures of Soda Ash and Blue Vitriol, Estimation of, by Titration with Potassium Hydroxide, 340
——— and Zinc, Electro-Analytical Estimation of, Application of Tantalum Electrodes to, 481
——— as Sulphide, Estimation of, 582
——— Compounds or Bismuth, and Formaldehyde, Interaction between, in the Presence of Alkali, 66
———, Electrolytic Separation of, from Tungsten and Molybdenum, 224
———, Estimation of, by Means of Sodium Hypophosphite, 75, 519, 520
———, Group, the Metals of the, Quantitative Volatilisation and Separation of Phosphoric Acid from, and from Aluminium, Tin, and the Alkali Metals, 525
———, in Burnt Pyrites, Rapid Estimation of, 389
——— in Cocoa and Chocolate, 145
——— in Distilled Water, 474
——— in Ores Containing Arsenic, Antimony, or Bismuth, Electrolytic Estimation of, 222
———, Thiocyanate-Permanganate Method for, 223
——— in Presence of Cadmium, Arsenic, Iron, or Lead, the Oxalate-Permanganate Method of Estimating, 122
——— in Solutions Containing Nitric Acid, Electrolytic Determination of, 340
——— in White Metal Alloys, Determination of, 222
———, Lead, and Zinc, in Supply-Water, Detection and Colorimetric Estimation of, 123
———, ———, Tin, and Antimony, Alloys of, Analysis of, 584
——— Tin Alloys, Analysis of, 388
———, Traces of, Detection of, 167
———, Zinc, and Nickel, Solutions of, Action of an Excess of a Soluble Ferrocyanide on, and a Volumetric Method for the Estimation of Nickel, 524
Cordite and Nitroglycerine (II.). Guncotton (I.). Theoretical (III.). Heat Test, 275
Corn, Kaffir, Colorimetric Method for the Determination of Hydrocyanic Acid in Plants, with Special Reference to, 569
Cotton, Free Sulphuric Acid in, Detection of, 510
——— Goods, Small Quantities of Wool in, Estimation of, 510
———, the "Degree of Bleaching" [Chemical Condition] of, Estimation of, 78

Cottonseed Foots, Total Fatty Acids in, Estimation of, 33, 34
——— - Meal and Wheat Bran, Phytic Acid in, 419
——— - Oil, Halphen's Test for, Modification of, 68
Coumarin in Melilotus, Estimation of, 145
Cow's Milk, Human Milk, and Buttermilk, the Physico-Chemical Behaviour of, Effect of Boiling on, 205
Cows Suffering from Foot and Mouth Disease, Milk from, Composition of, 328
Cream and Milk, Benzoic Acid in, the Detection and Estimation of, 555
———, the Estimation of Boric Acid in, a Quick and Accurate Method for, 140
-, Boric Acid and Fat in, Combined Estimation of, 141
Creatine, Creatinine, and Mono-amino Acids in Certain Fishes, Mollusca, and Crustacea, Estimation of, 26
Creolins, Phenols in, Estimation of, 325
Cryoscopic Solvent, Ethylene Dibromide as, 32
Crystalline Substances, Sections of, Preparation of 483
"Cupferron," Use of, in the Separation of Titanium from Aluminium, 390
(Cyanides and Ferrocyanides, Detection of) Berlin (Prussian) Blue Reaction, 111
Cyanogen Compounds in Gas Liquor, Estimation of, 31
Cyanogenesis under Digestive Conditions, 568

Demerara Sugar. Law Report, 595
Density and Internal Friction of Gases, Apparatus for Determining, 591
——— and Solution Volume of Some Proteins, 279
——— of Solids, Improved Pyknometer for, 130
Detonation, Velocity of, and Brisance, Estimation of, 464
(Dextrose) Sugar in Urine, Estimation of, 65
Di- and Tri-Phenols, New Colour Reactions of, 470
Diabetic Urine, Pentoses in, Detection of, 64
Dialysing Apparatus, 394
Dialysis, Application of, and Estimation of Oxidising Power as Aids in Judging Soils, 369
———, Quantitative, New Apparatus for, 232
Dichromate Ion, Electrometric Titration of, with Ferrous Iron, 585
———, Titration of Iron with, Application of Diphenylcarbazide ("Diphenylcarbohydrazide") as Indicator in, 167
Didymium, Thorium, Lanthanum, Cerium, Erbium, and Silica, Separation of Tungsten from, 481
"Difference Value," Polenske, for the Detection of Certain Animal Fats and its Theoretical Basis, 204
Diffusion of Dissolved Substances, New Method for Determining, 350
Digitalis Glucosides, Reactions of Digitonin, 265
——— Preparations, Physiological Standardisation of, 462
Digitonin. Reactions of Digitalis Glucosides, 265
Digitoxine, Reactions of, 457
Dilatometer and Specific Gravity Bottle, Combination, 485
Dimethyl Sulphate Test for Determining Small Amounts of Petroleum or Asphalt Products in Tars, Application of the, 277
Dinner, Anniversary, 96
Diphenylamine and Hydrochloric Acid, Behaviour of Certain Sugars with, 435
Diphenylcarbazide ("Diphenylcarbohydrazide") as Indicator in Titration of Iron with Dichromate, Application of, 167
Disinfectant Value of Mercuric Hydroxycyanide, 461
Disinfectants, Method of Standardising with and without Organic Matter, 422

Disinfectants, the Bacterial Testing of: A Practical Criticism, 190
Dissolved Oxygen in Water, the Determination of, Apparatus for, 396
——————————, Practical Field Method for, 397
——, the Estimation of, Use of Sodium Potassium Tartrate in, 442
— Substances, the Diffusion of, New Method for Determining, 350
α-Distearin and β-Palmitodistearin, the Synthetic Preparation of, and Nomenclature of Mixed Glycerides, 214
Distillation, Destructive, Volatile Products of, Estimation of Water in, 348
————, Direct Estimation of Water in Foods, etc., by, 207
———— of Coal-Tar, Residues from, Detection of Natural Asphaltum and Petroleum Pitch in, 157
————, Quantitative Ammonia, by Aeration. (III.), 580
Distilled Vinegar, Adulteration of Cider Vinegar with, Detection of, 565
——— Water, Copper in, 474
——— Wool Fat, 106
Dressings, Textile, Gelatin in, Detection of, 332
Drying Rates of Raw Paint Oils, 468
Dumas' Method, Nitrogen Estimations by, Absorption Apparatus for Use in, 234
Dura, Sudan, 323
Dust in Gas, Apparatus for Estimating, 48
Dusts, Coal, Relative Inflammabilities of. Part IV. Volatile Constituents of Coal, 512
Dyes, Certain Acid Coal-Tar, Mixtures of, Quantitative Separation of, 112
——, Subsidiary, in the Permitted Food Colours, Quantitative Separation and Determination of, 375
——, the Estimation of, Methods for, Comparative Investigation of, 113
Dyestuffs, Aniline [Magenta], in Foods, and Especially in Wines, New Reaction for the Detection of, 322
————, Small Amounts of, Identification of, by Oxidation with Bromine, 376
Dynamite and Black Powder, Analysis of, 514

Earthnut (Arachis) Oil in Olive Oil, Detection of, by the Franz-Adler Method, 58
———— Oil, Nitrobenzene in, Estimation of, 457
Earths, Alkaline, Colour Reactions of, with Oxygallol Derivatives, 473
East-Indian Tanned Hides, Various, the Analysis of, 363
Ebullioscope of High Accuracy, 482
Effluents, Colloids in, Estimation of, 269
————, Sewage, the Absorption of Oxygen by, Measurement of, 123
Eggs, Pastry Containing, Analysis of, 417
Egyptian Butter and Samna, 242
Electric Resistance Furnace. Application of, to Determination of Oxygen in Iron and Steel, 168
Electrically Heated Apparatus for Measuring Rotatory Power at Constant Temperature, 300
Electro-Analysis, Development of, 341
—————— with Light Platinum Electrodes, 43
Electrochemical Analysis, Small Apparatus for, 179
—————— Indicator for Oxidising Agents, 353
Electrodes, Light Platinum, Electro-Analysis with, 43
————, Tantalum, Application of, to Electro-Analytical Estimation of Copper and Zinc, 481
Electrolytic Deposition of Antimony, 581

Electrolytic Determination of Copper in Solutions Containing Nitric Acid, 340
——— Estimation of Copper in Ores Containing Arsenic, Antimony, or Bismuth, 222
——— Separation of Copper from Tungsten and Molybdenum, 224
——— of Lead, Influence of Colloids on, 76
Electrometric Titration of Dichromate Ion with Ferrous Iron, 585
Embelic Acid, Microscopical Detection of, 559
Emulsification in Extractions, Prevention of, by Immiscible Solvents, 48
Engler's and Redwood's Viscosimeters, Comparison of, 398
Enzyme, the Catalase, of Milk, and Simple Method for Determining the Fermentative Activity of Milk and Various Feeding-Stuffs, 328
Enzymes of the Tobacco Plant, 506
———, Precipitation of, from their Solutions by Means of Moist Aluminium Hydroxide, 423
Erbium, Thorium, Lanthanum, Cerium, Didymium, and Silica, Separation of Tungsten from, 481
Essential Oil of Cocoa, 20
——— of Jamaica Ginger, 56
——— Oils, Alcoholic Solutions of, and in Absinthe and Similar Liqueurs, Essential Oils in, Estimation of, 418
——— from Various Countries, 559
———, Glyceryl Acetate in, Estimation of, 148
———, Oxidation Assay of, 69
Esters, Cellulose, Non-Inflammable, Analysis and Examination of. Acetylcelluloses, 577
———, Isomeric, the Fatty Acids and their, the Absorption of Ultra-Violet Rays by, Quantitative Study of, 113
———, Other Aliphatic Alcohols and their, and Amyl Alcohol, Detection of, 574
Ethane, Methane, and Propane, Mixtures of Hydrogen with, Analysis of, 121
———, ———, Propane, and Isobutane, Mixtures of Hydrogen with, Analysis of, 276
Ether, Testing, 560
Ethyoxyl and Methyoxyl Estimations, Volumetric Method for, 430
Ethyl and Methyl Alcohols in the Presence of Each Other, Estimation of, 330
———, Small Quantities of, in Aqueous Solutions, Estimation of, 575
Ethylaniline and Methylaniline, Colour Reaction of Hypochlorites with, 476
Ethylene Dibromide as Cryoscopic Solvent, 32
Evaporation, Continuous, of Large Volumes of Liquid under Diminished Pressure, Apparatus for, 591
——— Test for Mineral, Lubricating, and Transformer Oils, 333
Ewers' Process, the Estimation of Starch by, Apparatus for Use in, 445
Explosives, High, the Effective Strength of, Methods for the Determination of, 378
———, the Stability of, Unification of the Testing Methods of, International Commission for, Final Report (July 25, 1912), 377
Extract, Dry, of Wines and Fermented Liquors, Accurate Estimation of, 566
———, Meat, and Fresh Meat, Succinic Acid in, Occurrence of, 508
———, and Urine, Precipitation of Purine Bases from, by Means of Zinc Salts, 572
Extracting Liquids Decomposed by Heat, Apparatus for, 444
Extraction Apparatus, a Simple All-Glass, 143
——— for Solids and Liquids, 351
———, Simple, 49

Extraction with Liquefied Gases, 233
Extractions, Emulsification in, Prevention of, by Immiscible Solvents, 48
Extractive Matters, Non-Nitrogenous, in Fodder and Foodstuffs, 560
Extracts of Drugs Containing Oxymethylanthraquinones, Aloes in, Detection of, 321
—, Plant, Methods of Estimation of Carbohydrates, Especially in, 504
—, Soil, Fertilisers, and Plant Ashes, Potassium in, Estimation of, 47
—, Vanilla, Authentic, Chemical Composition of, together with Analytical Methods and Limits of Constants, 206
Extractum Ferri Pomati, Iron in, Estimation of, 149
——— *Pomatum* and *Ferri et Quininæ Citras*, Direct Iron Estimation in, 562

Fæces, Skatol and Indol Together in, Estimation of, 30
Farup and Schumacher-Jung Methods for the Estimation of Mercury in Urine, Comparison of, 111
Fat and Boric Acid in Cream, Combined Estimation of, 141
—, Butter, and its Substitutes, Viscosity of, 458
—, Colostrum, and Colostrum, Composition of, 107
—, Estimation by the Kumagawa-Suto Method, Oxidation of Fatty Acids and Unsaponifiable Matter during Process of, 432
— in Cheese and Milk Products, the Estimation of, Acid-Butyrometric Method for, 145
———, Estimation of, 417
———, ———, by the Acid-Butyrometric Method without the Use of Amyl Alcohol, 146
———, ———, by the "Neusal" Method, 146
— in Ice Creams, Cereals, and Chocolate, Rapid and Accurate Method for Determining, 558
— in Sweetened Ice Cream, Modified Babcock Test for, 323
—, Wool, Distilled, 106
—, ——, Oleins of, Detection of Mineral Oils in, 35
—, ——, Stearin of, 37
Fats and Oils, Other, Marine Animal Oil (Thran) in, Detection of, 35
——, Animal and Vegetable, Differentiation of, 458
——, ———, Certain, the Detection of, Polenske "Difference Value" for, and its Theoretical Basis, 204
——, the Reichert-Meissl Value of, Influence of Antiseptic Agents on, 68
Fatty Acid Mixtures, Fractional Precipitation of. (Detection of Rape Oil), 114
—— Acids and their Isomeric Esters, the Absorption of Ultra-Violet Rays by, Quantitative Study of, 113
———— in Soaps, Estimation of, Rapid Volumetric Method for, 432
———— of Kaya Oil, 458
————, the Lower, Analysis of Mixtures of, 273
————, Total, in Cottonseed Foots, Estimation of, 33, 34
————, Volatile, Detection of, 274
—— Oils, Hexabromide Number of, and the Estimation of Rape Oil in Raw Linseed Oils, 158
Feeding-Stuffs and Manures, Phosphorus in, Estimation of, 563
————, Various, and Milk, the Fermentative Activity of, Simple Method for Determining, and the Catalase Enzyme of Milk, 328
Fermentation, Mashing, and Boiling of Wort, Transformation of the Proteins during (III.). Nitrogenous Substances in Barley and Malt, 496
Fermented Liquors and Wines, the Dry Extract of, Accurate Estimation of, 566
Fern, Male, Extract of, 428

Fern, Male, Extract of, Analytical Constants of, 429
Ferri et Quininæ Citras and *Extractum Ferri Pomatum*, Direct Iron Estimation in, 562
Ferric Salts, Nitrous Acid in the Presence of, Detection of, 289
Ferro-Alloys and Steel, Total Carbon in, Estimation of, by Combustion in Oxygen under Pressure, 285
Ferrocyanide, a Soluble, Action of an Excess of, on Solutions of Zinc, Copper, and Nickel, and a Volumetric Method for the Estimation of Nickel, 524
(Ferrocyanides and Cyanides, Detection of), Berlin (Prussian) Blue Reaction, 111
Ferro-Silicon, Analysis of, 76
Ferrotitanium, Analysis of, 474
Ferrotungsten, Metallic Tungsten Powder, Tungsten Oxide, and Tungstic Acid, Determination of Phosphorus in, by Direct Solution, 290
Ferrous Iron, Electrometric Titration of Dichromate Ion with, 585
——— Sulphate, Monohydrated, and its Employment in Volumetric Analysis, 284
Ferrovanadium and Vanadium Steel, Phosphorus in, Determination of, 587
Fertilisers, Application of Folin's Method for the Determination of Ammonia to, 381
———, Potash, Perchlorate in, Estimation of Potassium as, 124
———, Soil Extracts, and Plant Ashes, Potassium in, Estimation of, 47
Fibre, Crude, and Cellulose, Estimation of, 415
Fibres, Kapok and Akon, Waxes and Resins of, 580
Field Method, Practical, for the Determination of Dissolved Oxygen in Water, 397
Filter-Beds and Springs, the Testing of, New Rapid Method for the Bacteriological Examination of Water and its Application for, 30
Fish and Vegetable Oil Mixtures, Properties of (III.). Studies on Fish Oils, 70
—— Roe as Human Food, 508
Fleischmann Formula, Table of Calculated Dry Substance of Milk by the, 324
Flock, Rag, Chlorine in, Estimation of, 582
———, ——, ———, Note on the Estimation of, 409
Flour, the Quality of, Estimation of, 55
———, ———, ——— Soluble Nitrogenous Substances as a Criterion of, 148
Flue Gas, the Exact Analysis of, Apparatus for, 351
Fluids and Organs, Body, *d*-Lactic Acid in, Estimation of, 569
Fluorine in Zinc Ores, Estimation of, 475
———, Volumetric Estimation of, 521
Fodder and Foodstuffs, Non-Nitrogenous Extractive Matters in, 560
Folin's Method for the Determination of Ammonia, Application of, to Fertilisers, 381
FOOD AND DRUGS ANALYSIS, ABSTRACTS, 19, 55, 105, 145, 204, 264, 321, 365, 414, 456, 496, 558
Food Colours, the Permitted, Subsidiary Dyes in, Quantitative Separation and Determination of, 375
—— -Poisoning and Food Infections, Bacterial, Report to the Local Government Board on, 535
—— Standards and Regulations: The Health Act, 1911. Western Australia (Report), 179
Foods and Other Organic Substances, Moisture in, Determination of, 177
———, Aniline Dyestuffs [Magenta] in, and Especially in Wines, New Reaction for the Detection of, 322
———, Canned, the Estimation of Tin in, Iodine Titration Method for the Estimation of Tin, Especially in Connection with, 23
———, etc., Water in, Direct Estimation of, by Distillation, 207
———, Formaldehyde in, Detection of, 497
———, Inspectors of, the Work of, for the Year 1911-1912, Report on, 131

Foods, Saccharin in, Estimation of, 326
Foot and Mouth Disease, Cows Suffering from, Composition of Milk from, 328
Foots, Cottonseed, Total Fatty Acids in, Estimation of, 33, 34
Formaldehyde and Bismuth or Copper Compounds, Interaction between, in the Presence of Alkali, 66
———— and Methyl Alcohol, Simultaneous Estimation of, 576
———— and Nitrous Acid, Interaction between, 67
————, Detection of, with *p*-Nitrophenylhydrazine, 466
———— in Foods, Detection of, 497
———— in the Juices of Plants, Occurrence of, 152
————, Saffron Containing, 420
————, the Milk Tests for, Interference of Hydrogen Peroxide with, 21
————, Total, in Fumigators and Commercial Solutions, Estimation of, 497
Formic Acid, Detection and Estimation of, 378
——— Acid as a Solvent, 513
——— and Acetic Acids, Detection of, 275
Fractional Precipitation of Fatty Acid Mixtures (Detection of Rape Oil), 114
Fractionation of California Petroleum by Diffusion through Fuller's Earth, 469
Fractures of Steel, Preparing Sections of, for Microscopic Examination, 528
Franz-Adler Method, Detection of Earthnut (Arachis) Oil in Olive Oil by the, 58
Freezing-Point of Milk, 368
Friction, Internal, and Density of Gases, Apparatus for Determining, 591
Frog and Tortoise, the Blood of, the Estimation of Sugar in, Sources of Error in, 463
Fruit Juices, Salicylic Acid in, Detection and Estimation of, 420
Fuels, Especially Petroleum Products, Sulphur in, Determination of, 38
———, Liquid, Chemical Examination of, 212
Fuller's Earth, Fractionation of California Petroleum by Diffusion through, 469
Fumigators and Commercial Solutions, Total Formaldehyde in, Estimation of, 497
Funnel, New Separating, 234
Funtumia Elastica and *Manihot Ceara*, the Oils from, Examination of, and a Comparison of their Properties with those of Linseed and *Hevea* Oils, 259
Furnace, Electric Resistance, Application of, to Determination of Oxygen in Iron and Steel, 168
Fusel Oil, Valuation of, 66

Galactan, the Estimation of, Method for, 158
Gallic Acid and Tannin, Colour Reaction for, 467
Gas Analysis Apparatus, 395, 594
———— Laboratories at Coal Mines, Apparatus for, 482
Gas, Dust in, Apparatus for Estimating, 48
——, Flue, the Exact Analysis of, Apparatus for, 351
—— Fuels, Suction, Rapid Method of Testing, for Liability to Clog Gas Engine Valves, 332
—— Generator, Simple Gasoline, for Sulphur Determinations, 592
——, Hydrogen, Nitrogen, and Methane in, Determination of, by Combustion in a Quartz Tube, 341
——, Illuminating, Hydrogen Sulphide in, Apparatus and Method for Determining, 396, 593
——, Illuminating, the Estimation of Sulphur in, Volumetric Method for, 228
——, ————, the Testing of, Estimation of Sulphate in Ammonium Sulphate Solution, with Special Reference to, 382
——, Liquor, Cyanogen Compounds in, Estimation of, 31

Gas, Natural, the Estimation of, for the Production of Gasoline, Methods for, 577
—— Thermo-Regulators, Improvement in, 351
Gaseous Hydrocarbons, Hydrocarbons of the Acetylene and Olefine Series in Mixtures of, Estimation of, 158
Gases, Liquefied, Extraction with, 233
——, the Density and Internal Friction of, Apparatus for Determining, 591
Gasoline Gas Generator, Simple, for Sulphur Determinations, 592
——, the Production of, the Estimation of Natural Gas, Methods for, 577
Gasometric Estimation of Nitric Oxide and Oxygen, Depending on the Formation of Nitrous Anhydride, 391
—— Method, New, for the Estimation of Nitric Oxide: I., 45
Gay-Lussac Method of Silver Determination, 228
Gelatin in Textile Dressings, Detection of, 332
——, Studies on the Chemistry of, and its Estimation, 67
Gelatinising Temperatures of Starch Granules, Researches on, 74
German Pharmacopœia, the Methods of, Examination of Turpentine Oil by, 501
—— Regulations, New, for the Official Examination of Inks, 213
Ginger, Jamaica, Essential Oil of, 56
Glass, Certain Sorts of, Action of Baryta Water on, 118
Globulin, Casein and Albumin in Milk: Nephelometric Determination of Proteins, 571
Glucose, Small Amounts of, in Urine, Estimation of, 463
Glucosides, Digitalis, Reactions of. Digitonin, 265
Glues, the Jelly-Strength of, Improved Apparatus for Testing, 231
Glycerides, Mixed, Nomenclature of, and the Synthetic Preparation of α-Distearin and β-Palmitodistearin, 214
——, Mixed, of Palmitic and Stearic Acids, Present in Lard, 204
Glycerin-Jelly, the Use of, in Mounting Microscopical Objects, 407
Glycerol in Sweet Raisin Wines, Estimation of, 24
—— in Wines, Estimation of, 368
——, New Method of Estimating, 275
Glyceryl Acetate in Essential Oils, Estimation of, 148
Glycogen in Liver, Estimation of, 63
Glycyrrhizin in Sweets and in Liquorice, Estimation of, 367
Glyoxylic Reaction, Colour Reactions of Certain Indole Derivatives, and their Significance with Regard to the, 276
Gold and Platinum, Detection of, 476
—— Bromide Test for Semen, 29
—— Coast, Crude Petroleum from, 162
——, Platinum, Rhodium, and Iridium, Separation of Palladium from, 524
—— Salts, Delicate Colour Reactions of, 54
——, Volumetric Determination of, 390
Goldenberg Method (for Commercial Tartrates), 1898 and 1907, 435
Grains, Oil in, the Determination of, Rapid Control Method for, 57
Granules, Starch, the Gelatinising Temperatures of, Researches on, 74
Grapes and Wines, Estimation of Pentoses and Methylpentoses in; Investigations on Lead Precipitates from Wines, 25
Greases, Lubricating, Soaps in, Estimation of, 162
"Green Butter" and Cocoa Butter, a Simple Test for Differentiating between, 201
Green Plants, Respiratory Quotients of, 152
Groups, Carboxyl, in Organic Acids, Determination of, Improved Apparatus for, 299
Guaiacum Test for Bloodstains, the Value of the, 186
I.—Guncotton; II.—Nitroglycerine and Cordite; III.—Theoretical. Heat Test, 275

Hæmolysis, Detection and Differentiation of Poisonous and Non-Poisonous Saponins by, 151
Halogen Salts, Bromine in, Iodic Acid Method of Estimating, 166
————, the Estimation of Bromine in, Preparation of Selenic Acid and Sodium Selenate as Reagents for, 295
Halphen's Test for Cottonseed Oil, Modification of, 68
Hardened Marine Animal Oils, 373
———— Oils, Analysis of, 432
"Hardened" Oils, Note on, 102
Hardened Whale Oil, Colour Reactions of, 433
Hardness of Water, the Determination of, and Behaviour of Calcium and Magnesium Salts with Soap Solutions, 436
Heat Evolved by Bacteria, Micro-Calorimeter for Measuring, 421
——, Liquids Decomposed by, Apparatus for Extracting, 444
—— of Combustion and Elementary Analysis, Use of Bomb Calorimeter for, 533
—— Test. I.—Guncotton. II.—Nitroglycerine and Cordite. III.—Theoretical, 275
Heavy Metals, Salts of, Behaviour of Salts of Cæsium and Rubidium towards, 283
Hevea and Linseed Oils, Examination of the Oils from *Manihot Ceara* and *Funtumia Elastica*, and a Comparison of their Properties with those of, 259
Hexabromide Number of Fatty Oils and the Estimation of Rape Oil in Raw Linseed Oils, 158
Hickory Nuts and Hickory Nut Oil, 498
Hides, Tanned, Various East-Indian, the Analysis of, 363
Hiding Power and Opacity of Pigments, Tests on, 161
Higher Alcohols in Spirits, Estimation of, 564
———— in Wine, Estimation of, 23
Histidine, Detection of, 114
————, the Estimation of, Colorimetric Method for, 578
Hoffmann's Apparatus, Estimation of Water in Vegetables by, 177
Honey, Composition of, 265
———, Examination of, 20
———, Russian, Composition of, 149
Hop Analysis, 562
——, the Composition of the, Effect of Kiln-Drying at 140° F. on, 20
Hops, Constituents of, 459
——, Grown in Different Parts of the World, the Composition of, Comparative Study of, 366
——, Nitrogenous Constituents of, 459
Horseflesh, Carnosine, Methylguanidine, and Carnitin in, Occurrence of, 508
Human Bile, the Analysis of, 208
——— Milk, Cow's Milk, and Buttermilk, the Physico-Chemical Behaviour of, Effect of Boiling on, 205
Humus, Nitrogen in Contribution to Methods of Determining, 163
———, the Official Method of Determining, Modification of, 115
Hydriodic Acid in Presence of Phenol, Methoxyl Estimations with, 33
Hydrocarbons, Gaseous, Hydrocarbons of the Acetylene and Olefine Series in Mixtures of, Estimation of, 158
Hydrochloric Acid and Diphenylamine, Behaviour of Certain Sugars with, 435
Hydrocyanic Acid, Detection of, 332
———— in Plants, the Determination of, with Special Reference to Kaffir Corn, Colorimetric Method for, 569,
Hydrofluoric Acid Solution, Potassium Chloride in, Separation of Niobium and Tantalum by Means of, 226

Hydrogen Ion Concentration of Sea-Water, Colorimetric Estimation of, "Salt Error" in, 394
——— ———, Red Cabbage Extract as Indicator for Measuring, 114
———, Mixtures of, with Methane, Ethane, and Propane, Analysis of, 121
———, ———, Propane, and Isobutane, Analysis of, 276
———, Nitrogen, and Methane in Gas, Determination of, by Combustion in a Quartz tube, 341
——— Peroxide Colour Method, Estimation of Vanadium in Steel by the, 589
——— ———, Interference of, with the Milk Tests for Formaldehyde, 21
——— Sulphide in Illuminating Gas, Apparatus and Method for Determining, 396, 593
——— ——— in Natural Waters, Small Quantities of, Estimation of, 532
——— Table, New, 167
(Hydrogenised) Solidified Oils and the Detection of Arachidic Acid, 160
Hydrogenising Apparatus, Skita's, Simple Modification of, 484
Hydrolysis of Lævulosans and its Application for Analytical Purposes, 429
——— of Organic Phosphorus Compounds by Dilute Acid and Dilute Alkali, 278
———, Tryptic Protein, Measurement of, by Determination of the Tyrosine liberated, 153
Hydrolysts, Invertase and Acid as, Determination of Sucrose in Cane Molasses by the Double Polarisation Method, Using, 281
Hydrolytic Action a Source of Error in the Estimation of Iodine and Bromine in Mineral Waters, etc., 532
Hydrometers, Standardisation of, 395
Hydrosulphite in Volumetric Analysis [of Dyestuffs], Use of,
Hydrosulphites, Analysis of, 284
Hydroxides and Carbonates of Potassium and Sodium when Together in Solution, Estimation of, 166
Hydroxycyanide, Mercuric, Disinfectant Value of, 461
Hydroxyl Ions, the Concentration of, New Method for the Determination of, 522
Hydroxylamine, Estimation of, 437
Hypochlorites, Colour Reaction of, with Methylaniline and Ethylaniline, 476
———, Estimation of, 224
———, Method of Analysing, 521
Hypophosphite, Sodium, Estimation of Copper by Means of, 75, 519, 520
Hypophosphites and Hypophosphorous Acid, Volumetric Estimation of, 342
———, Estimation of, 438
Hypophosphorous Acid, Separation of Thorium from Monazite Sand by Means of. Detection of Cerium, 443

Ice Cream, Sweetened, Fat in, Modified Babcock Test for, 323
— Creams, Cereals, and Chocolate, Fat in, Rapid and Accurate Method for Determining, 558
Illuminating Gas, Hydrogen Sulphide in, Apparatus and Method for Determining, 396, 593
———, the Estimation of Sulphur in, Volumetric Method for, 228
———, the Testing of, Estimation of Sulphate in Ammonium Sulphate Solution, with Special Reference to, 382
Immiscible Solvents, Prevention of Emulsification in Extractions by, 48
India, Lemon-Grass Oils from, 160
———, Turpentine Oils from, 165
Indican Reaction, New, 508, 578
Indicator, an Electrochemical, for Oxidising Agents, 353

Indicator for Measuring Hydrogen Ion Concentration, Red Cabbage Extract as, 214
—— in Titration of Iron with Dichromate, Application of Diphenylcarbazide ("Diphenylcarbohydrazide") as, 167
—— Method for the Estimation of the Permeability of Cells by Alkalis, 327
——, New, 427
Indol and Skatol together in Fæces, Estimation of, 30
—— Derivatives, Certain, Colour Reactions of, and their Significance with Regard to the Glyoxylic Reaction, 276
Inks, the Official Examination of, New German Regulations for, 213
INORGANIC ANALYSIS, ABSTRACTS, 40, 75, 116, 166, 220, 283, 338, 381, 436, 473, 517, 580
INSTITUTE OF CHEMISTRY, 51, 86, 182, 240, 406.
Institute of Metals (Ghent Meeting), 304
Insulated Wires, Caoutchouc in Rubber Wares, Particularly, Estimation of, 111
Invert Sugar [Commercial], Detection of, by Means of β-Naphthol, 217
Invertase and Acid as Hydrolysts, Determination of Sucrose in Cane Molasses by the Double Polarisation Method, Using, 281
Iodate, Action of Persulphate on, and Estimation of Periodate in Presence of Persulphate, 479
Iodic Acid Method of Estimating Bromine in Halogen Salts, 166
Iodide, Sodium, Purification of Acetone by Means of, 424
Iodimetric Estimation of Free and Combined Chromic Oxide, 386
—————— of Iron, 586
—————— of Persulphuric Acid, 293
Iodine and Bromine in Mineral Waters, etc., the Estimation of, Hydrolytic Action a Source of Error in, 532
—— in Organic Substances, Estimation of, 427
——, the Recovery of, from Residues, 409
—— Titration Method for the Estimation of Tin, Especially in connection with the Estimation of Tin in Canned Foods, 23
Iodoform, Carbon Tetriodide in the Presence of, Estimation of, 373
Ion Concentration, Hydrogen, Indicator for Measuring, Red Cabbage Extract as, 214
————, ————, of Sea-Water, "Salt Error" in Colorimetric Estimation of, 394
Ion, Dichromate, Electrometric Titration of, with Ferrous Iron, 585
Ions, Hydroxyl, the Concentration of, New Method for the Determination of, 522
Iridium, Gold, Platinum, and Rhodium, Separation of Palladium from, 524
Iron Alloys, Steel, and Cast Iron, Total Carbon in, Rapid Determination of, 442
—— and Chromium, Quantitative Separation of, 387
—— and Copper, Estimation of Palladium and its Separation from, 79
—— and Manganese, Separation of, 121
—— and Steel, Carbon in, Rapid Method for Estimating, 384
————, Colorimetric Method for Titanium in, 588
————, Determination of Oxygen in, Application of Electric Resistance Furnace to, 168
————, Phosphorus in, Estimation of, 291
————, the Estimation of Phosphorus in, without Separation of Silicon, Reduction of Manganese Dioxide by Nitrous Acid, and the Use of this Reaction in, 77
——, Cadmium, Arsenic, or Lead, Copper in Presence of, the Oxalate-Permanganate Method for Estimating, 122
——, Chromium, and Vanadium, in Presence of One Another, the Volumetric Estimation of, a New Method for, 99

Iron, Chromium in Presence of, Volumetric Estimation of, 387
—— Estimation, Direct, in *Ferri et Quininæ Citras* and *Extractum Ferri Pomatum*, 562
——, Ferrous, Electrometric Titration of Dichromate Ion with, 585
——, Hydrated Oxide of, Valuation of, by Means of the "Sulphuretted Hydrogen Burette," 522
—— in *Extractum Ferri Pomati*, Estimation of, 149
—— in *Syr. Ferri Phosph. Co.* and Other Pharmaceutical Preparations, the Estimation of, 447
—— in Water, Estimation of, 174
—— Iodimetric Estimation of, 586
——, Manganese in the Presence of, Estimation of, 225
——, Titanium in Presence of, Estimation of, 229
——, ———— in the Presence of, Estimation of, 82
——, Titration of, with Dichromate, Diphenylcarbazide ("Diphenylcarbohydrazide") in, Application of, 167
——, Traces of, in Cane and Beet Sugar Factory and Refinery Products, Estimation of, 59
Isobutane, Methane, Ethane, and Propane, Mixtures of Hydrogen with, Analysis of, 276

Jamaica Ginger, Essential Oil of, 56
———— Rum, Method of Distinguishing, from Artificial Rum, 368
Japan and China, "Wood Oils" from, 580
Jelly-Strength of Glues, Improved Apparatus for Testing. 231
Juices, Fruit, Salicylic Acid in, Detection and Estimation of, 420
———— of Plants. Formaldehyde in, Occurrence of, 152

Kaffir Corn, Colorimetric Method for the Determination of Hydrocyanic Acid in Plants, with Special Reference to, 569
Kapok and Akon Fibres, Waxes and Resins of, 580
———— Seeds and Kapok Oil, 467
Kaya Oil, Fatty Acids of, 458
Kjeldahl-Bertrand Method, Estimation of Sugars by the, 218
————-Gunning Method, Source of Error in the, 467
Knorre, G. von, the Method of, Estimation of Manganese in Castings and Steels by, 286
Köttstorfer Value for the Estimation of Resins and Substitutes in Caoutchouc Articles, Use of, 337
Kumagawa-Suto Method, Process of Fat Estimation by the, Oxidation of Fatty Acids and Unsaponifiable Matter during, 432

Lactic Acid, Estimation of, 379
d-Lactic Acid, in Body Fluids and Organs, Estimation of, 569
Lactic Acid in Urine, Estimation of, 328, 370
————————, Tartaric, Citric, and Malic Acids in Musts, Wines, etc., Estimation of, 504
Lactose and Casein in Milk, Estimation of, 21
———— in Milk, Estimation of by Precipitation with Ammonium Sulphate, 418
————————, the Estimation of, Aluminium Hydroxide as a Protein Precipitating Reagent in, 418
————, the Polarimetric Estimation of, Influence of Ammonium Sulphate on, 217
Lævulosans, Hydrolysis of, and its Application for Analytical Purposes, 429
Lamb and Mutton, New Zealand, Fresh and Frozen, Chemical and Bacteriological Study of, 27
Lanolin, Paraffins of, Detection of, 458

Lanthanum, Thorium, Cerium, Erbium, Didymium and Silica, Separation of Tungsten from, 481
Lard, Palmitic and Stearic Acids Present in, Mixed Glycerides of, 204
Law Report. (Demerara Sugar), 595
Lead, Action of Various Waters on, 530
—— Alloys, Analysis of, and Cathodic Estimation of Lead, 582
—— Cadmium, Arsenic, or Iron, Copper in Presence of, the Oxalate-Permanganate Method for Estimating, 122
——, Cathodic Estimation of, and Analysis of Lead Alloys, 582
—— Chromate and Lead Sulphide, Solubility of, 476
——, Copper, and Zinc, in Supply-Water, Detection and Colorimetric Estimation of, 123
——, Nickel, and Zinc, Estimation of, by Precipitation as Oxalate and Titration with Permanganate, 122
—— Peroxide in Minium, Rapid Method of Estimating, 343
—— Precipitates from Wines, Investigations on; Estimation of Pentoses and Methylpentoses in Grapes and Wines, 25
——, Small Quantities of, Estimation of, 440
—— Sulphate, Determination of, and the Ultimate Estimation of "Sublimed White Lead" in Rubber, 44
—— Sulphide and Lead Chromate, Solubility of, 476
——, the Electrolytic Separation of, Influence of Colloids on, 76
——, Tin, Antimony, and Copper, Alloys of, Analysis of, 584
Leather, Free Sulphur in, Estimation of, 467
Lecithin, Phosphorus in, Estimation of, 58
—— Preparations and the Estimation of Lecithin, 205
Lehmann's Method, Estimation of Reducing Sugars by, 219
Lemon-Grass Oils from India, 160
Liebig Combustion Method, Modification of the, 301
Lime-Magnesia Ratio in Soil Analysis, Significance of, 126
Linseed and *Hevea* Oils, Examination of the Oils from *Manihot Ceara* and *Funtumia Elastica* and a Comparison of their Properties with those of, 259
—— Oil, Adulteration in, the Detection of, 3, 104
—— ——, Effect of Certain Pigments on, Manganese Content of Raw Linseed Oil, 215
—— ——, Oxygen Absorption Test for, 159
—— Oils, Raw, the Estimation of Rape Oil in, and Hexabromide Number of Fatty Oils, 158
Lipoids in Blood-Serum, Estimation of, 28
Liquefied Gases, Extraction with, 233
Liqueurs, Similar, and Absinthe, Estimation of Essential Oils in, and in Alcoholic Solutions of Essential Oils, 418
Liquid Fuels, Chemical Examination of, 212
——, Large Volumes of, the Continuous Evaporation of, under Diminished Pressure, Apparatus for, 591
Liquids and Solids, Extraction Apparatus for, 351
—— Decomposed by Heat, Apparatus for Extracting, 444
——, Organic, Determination of Chlorides and Bromides in, 211
Liquor, Gas, Cyanogen Compounds in, Estimation of, 31
Liquors, Fermented, and Wines, the Dry Extract of, Accurate Estimation of, 566
Liquorice and Sweets, Glycyrrhizin in, Estimation of, 367
Lithium, Determination of, 168
—— in Mineral Waters, Estimation of, 531

Lithopones, Analysis of Paints, and Especially, 334

Liver, Glycogen in, Estimation of, 63

Lloyd's Reagent, Precipitation of Alkaloids by, 426

Lubricating Greases, Soaps in, Estimation of, 162

————, Mineral, and Transformer Oils, Evaporation Test for, 333

———— Oils, Viscosity of, 36

Macagno's Method, Volumetric Estimation of Phosphorus in Steel by, 526

[Magenta] Aniline Dyestuffs in Foods, and Especially in Wines, the Detection of, New Reaction for, 322

Magnesia, Lime-, Ratio in Soil Analysis, Significance of, 126

Magnesium and Calcium Salts, Behaviour of, with Soap Solutions, and the Determination of Hardness of Water, 436

————, Calcium, Strontium, and Barium, Spectroscopic Detection of, 584

———— Carbonate, Asbestos Mixtures, etc., Magnesium Oxide in, Calorimetric Detection and Estimation of, 76

————, Sulphates, Tartaric Acid, and Calcium in Wine, Physico-Chemical Estimation of, 421

Male Fern, Extract of, 428

————, ————, Analytical Constants of, 429

Malic and Tartaric Acids in the Same Solution, Determination of, 381

——, Tartaric, and Succinic Acids in Wine, Estimation of, 502

——, ————, Citric, and Lactic Acids in Musts, Wines, etc., Estimation of, 504

Malt and Barley, Nitrogenous Substances in. III. Transformation of the Proteins during Mashing, Boiling of Wort, and Fermentation, 496

—— and Unmalted Cereals, American Beers Made from, Use of the Immersion Refractometer in Examining, 456

Manganese and Chromium, New Separation of, 474

————————, Separation of, 474

————————, Small Quantities of, in Minerals and Rocks, Estimation of, 226

———— and Iron, Separation of, 121

———— Content of Raw Linseed Oil. Effect of Certain Pigments on Linseed Oil, 215

———— Dioxide, Reduction of, by Nitrous Acid, and the Use of this Reaction in the Estimation of Phosphorus in Iron and Steel with Separation of Silicon, 77

———— in Castings and Steels, Estimation of, by the Method of G. von Knorre, 286

———— in Rocks, Slags, Ores, and Spiegels, Volumetric Determination of, 170

———— in Soil, Estimation of, 343

———— in the Presence of Iron, Estimation of, 225

————, Small Quantities of, in Water, Colorimetric Estimation of, 225, 477

-, Volumetric Estimation of, by the Volhard-Wolff Method. Chemikerkommission des Vereins deutscher Eisenhüttenleute, 391

Manihot Ceara and *Funtumia Elastica*, the Oils from, Examination of, and a Comparison of their Properties with those of Linseed and *Hevea* Oils, 259

Manketti Nuts, Oil of, 105

Manures and Feeding-Stuffs, Phosphorus in, Estimation of, 563

Maple Products, Analysis of. I.: Electrical Conductivity Test for the Purity of Maple Syrup, 499

Margarine, Vegetable Oils in, Detection of, 21

Marine Animal Oil (Thran) in Other Oils and Fats, Detection of, 35

———— Oils, Hardened, 373

Match Compositions, White Phosphorus in, Detection of, 289
Maumené Figure of Oil of Turpentine, 338
—— Test, Detection of Petroleum Products in Oil of Turpentine by Means of the, 70
Meat Extract and Fresh Meat, Succinic Acid in, Occurrence of, 508
———— and Urine, Precipitation of Purine Bases from, by Means of Zinc Salts, 572
Meats, Benzoic Acid in, Estimation of, 414
Mechanical Agitator, 590
Melilotus, Coumarin in, Estimation of, 145
Melnik (Bohemian) Wine, 326
Melting- and Solidifying Points of Mixtures of Stearic and Oleic Acids, 579
Melting-Point Apparatus for High Temperatures, 483
Mercuric Hydroxycyanide, Disinfectant Value of, 461
—— Oxide as a Standard for Volumetric Analyses, 439
Mercury as Metal, Estimation of, by the Dry Method, 227
—— in Textile Materials, Estimation of, 63
—— in the Air of Rooms, Etc., where the Metal is Used, Estimation of, 477
—— in Urine and Animal Tissues, the Estimation of, Method for, 153
————, the Farup and Schumacher-Jung Methods for the Estimation of, Comparison of, 111
——, Small Quantities of, in Organic Substances, Estimation of, 515
Metallurgical Calculations, Notes on, and on Valuation of Ores and Minerals, 171
Metals, Heavy, Salts of, Behaviour of Salts of Cæsium and Rubidium towards, 283
Methane, Ethane, and Propane, Mixtures of Hydrogen with, Analysis of, 121
——, ——, Propane, and Isobutane, Mixtures of Hydrogen with, Analysis of, 276
——, Hydrogen, and Nitrogen in Gas, Determination of, by Combustion in a Quartz Tube, 341
——, Preparation and Analysis of, 430
Methoxyl and Ethoxyl Estimations, Volumetric Method for, 430
—— Estimations with Hydriodic Acid in Presence of Phenol, 33
Methyl Alcohol and Formaldehyde, Simultaneous Estimation of, 576
————, Detection and Estimation of, 509
———— in Spirits, Estimation of, 56
————, Small Quantities of, in Blood and Animal Tissues, Estimation and Detection of, 62
————, the Identification of, New Method for, 379
—— and Ethyl Alcohols in Aqueous Solutions, Small Quantities of, Estimation of, 575
in the Presence of Each Other, Estimation of, 330
Methylaniline and Ethylaniline, Colour Reaction of Hypochlorites with, 476
Methylene Blue, Determination of Stannous Chloride and of Chlorates by Means of, 126
————, the Quantitative Reduction of, by Milk Bacteria, Investigations on, and the Use of this Dye for Determining the Condition of Milk, 62
Methylguanidine, Carnosine, and Carnitin in Horseflesh, Occurrence of, 508
Methylpentoses and Pentoses in Grapes and Wines, Estimation of; Investigations on Lead Precipitates from Wines, 25
Mett's Method for Determining the Activity of Pepsin and the Acidity-Maximum of Peptic Digestion, 29
Microbes, Pathogenic, in "Raw" River Water, and in Crude Sewage, Search for, 370

Micro-Calorimeter for Measuring Heat Evolved by Bacteria, 421
Microchemical Reagents, 83
Micropyrometer, 130
Microscopic Examination, Preparing Sections of Fractures of Steel for, 528
Microscopical Detection of Embelic Acid, 559
——————— Protoplasmalipoids, especially in Muscular Tissue, 423
——————— Objects, the Use of Glycerin-Jelly in Mounting, 407
Microsublimation of Alkaloids under Reduced Pressure, 426
Milk, Added Water in, Detection of, by Means of the Refractometer Value of the Serum, 500
—— and Cream, Benzoic Acid in, the Detection and Estimation of, 555
——, Albumin, Casein, and Globulin in: Nephelometric Determination of Proteins, 571
—— and Cream, the Estimation of Boric Acid in, a Quick and Accurate Method for, 140
——, and Various Feeding-Stuffs, the Fermentative Activity of, Simple Method for Determining, and the Catalase Enzyme of Milk, 328
—— Bacteria, Investigations on the Quantitative Reduction of Methylene Blue by, and the Use of this Dye for Determining the Condition of Milk, 62
——, Calculated Dry Substance of, by the Fleischmann Formula, Table of, 324
——, Casein, and Lactose in, Estimation of, 21
——, Cow's, Human Milk, and Buttermilk, the Physico-Chemical Behaviour of, Effect of Boiling on, 205
——, Evaporated, and Milk-Powders, Milk-Fat in, Rapid and Accurate Gravimetric Method for Determining, 147
——, Freezing-Point of, 368
—— from Cows Suffering from Foot and Mouth Disease, Composition of, 328
——, Lactose and Casein in, Estimation of, by Precipitation with Ammonium Sulphate, 418
——, ———— in, the Estimation of, Aluminium Hydroxide as a Protein Precipitating Reagent in, 418
——, Morres's "Alizarol" Test for, 460
——, Nitrates and Nitrites in, 450
——, Nitric Acid (Nitrates) in, Detection of, 563
——, Peroxydase Reaction of. (Influence of Preservatives, etc.), 325
——, Potassium Bichromate in, Detection of, 563
—— Products and Cheese, the Estimation of Fat in, Acid-Butyrometric Method for, 145
——, Proteins in, Estimation of, by Aldehyde Titration, 266
    Solubility of, in the Constituents of the Serum, 570
Samples for Analysis, Preservation of, 325
Tests for Formaldehyde, Interference of Hydrogen Peroxide with the, 21
, the Catalase Enzyme of, and Simple Method for Determining the Fermentative Activity of Milk and Various Feeding-Stuffs, 328
, the Composition of, 252
, the Distribution of Cell Elements in, Influence of Centrifugal Action on, 108
, the Specific Gravities of, Vieth's Table for Correcting, Extension of, 460
, the Vitamine Fraction of, Attempt to Estimate, 269
, Total Solids of, 21
Treated with Potassium Dichromate, Alteration of, 325
Mine Air, the Examination of, Apparatus for, 128
Mineral, Lubricating, and Transformer Oils, Evaporation Test for, 333
———— Oils in Oleins of Wool Fat, Detection of, 35
———— Powders, the Specific Gravity of, Determination of, 296

Mineral Waters, etc., Iodine and Bromine in, the Estimation of, Hydrolytic Action a Source of Error in, 532
———————, Lithium in, Estimation of, 531
Mineralogical Analysis of Soils, 125
Minerals and Ores, Valuation of, Notes on, and on Metallurgical Calculations, 171
——— and Rocks, Silicate, Water in, Estimation of, 47
———————, Small Quantities of Manganese and Chromium in, Estimation of, 226
Mines, Coal, Gas Analysis Laboratories at, Apparatus for, 482
Minium, Lead Peroxide in, Rapid Method of Estimating, 343
Miscibility Curves, Use of, in the Examination of Spirits of Camphor, 204
Moisture, Determination of, 174
——— in Foods and Other Organic Substances, Determination of, 177
——— in Organic Substances, Determination of, 177
Molasses, Cane, Sucrose in, Determination of, by the Double Polarisation Method, Using Invertase and Acid as Hydrolysts, 281
Molybdenum and Tungsten, Electrolytic Separation of Copper from, 224
——, Detection of, 523
——, Sensitive Reaction of, 478
Molybdic Acid, Sensitive Reaction for, 43
Monazite Sand, Separation of Thorium from, by Means of Hypophosphorous Acid. Detection of Cerium, 443
Mono-amino Acids, Creatine, and Creatinine in Certain Fishes, Mollusca, and Crustacea, Estimation of, 26
Morphine in Opium and Opium Preparations, Estimation of, 150
Multiviscosimeter, the, 178
Muscular Tissue, Protoplasmalipoids, Especially in, Detection of, 423
Mustard Powder, Black, 500
Musts, Wines, etc., Tartaric, Citric, Lactic, and Malic Acids in, Estimation of, 504
Mutton and Lamb, New Zealand, Fresh and Frozen, Chemical and Bacteriological Study of, 27
Mylius's Reaction for the Detection of Cholalic Acid and Other Biliary Acids, Method of Sensitising, 506

β-Naphthol, Detection of [Commercial] Invert Sugar by Means of, 217
Native Bitumens and their Residuals, Characteristics and Differentiation of, 371
Neodymium, Some Quantitative Separations of, 343
Nephelometric Determination of Proteins: Casein, Globulin and Albumin in Milk, 571
"Neusal" Method, Estimation of Fat in Cheese by the, 146
Neutral Red Paper as Indicator for Alkalimetry of Serum, 328
Nickel in Plated Articles, Detection of, 45
———, Lead, and Zinc, Estimation of, by Precipitation as Oxalate and Titration with Permanganate, 122
———, the Detection and Estimation of, by Means of α-Benzildioxime, 316
———, Zinc, and Copper, Solutions of, Action of an Excess of a Soluble Ferrocyanide on, and a Volumetric Method for the Estimation of Nickel, 524
(Ninhydrin) Triketohydrindene Hydrate, Colour Reactions of, 579
Niobium and Tantalum, Separation of, by Means of Potassium Chloride in Hydrofluoric Acid Solution, 226
Nitrate in Presence of Nitrite, Detection of, 170
Nitrates and Nitrites in Milk, 450
————, Determination of, by Phenol Disulphonic Acid Method, Influence of Chlorine on, 339
(Nitrates) Nitric Acid in Milk, Detection of, 563

Nitric Acid, Estimation of Cellulose by Means of, 509
——— in Sulphuric Anhydride, Qualitative Detection of, 78
———, Nitrous Acid in the Presence of, Detection of, 585
———, Solutions Containing, Electrolytic Determination of Copper in, 340
———, the Presence of, Colour Reactions of Oils with Certain Phenols in, 483
—— Oxide and Oxygen, Gasometric Estimation of, Depending on the Formation of Nitrous Anhydride, 391
———, Estimation of, 171
the Estimation of, New Gasometric Method for: I., 45
Nitrite, Detection of Nitrate in Presence of, 170
Nitrites and Nitrates in Milk, 450
———, Estimation of, by Means of Thiocarbamide, and the Interaction of Nitrous Acid and Thiocarbamide in Presence of Acids of Different Strength, 440
Nitrobenzene in Earthnut Oil, Estimation of, 457
Nitrogen and Sulphur, Water Formed on Burning Substances Containing, Estimation of, in a Calorimetric Bomb, 347
———, Elementary, Direct Estimation of, by Means of Commercial Calcium Carbide, 287
——— Estimations, by Dumas' Method, Absorption Apparatus for Use in, 234
———, Hydrogen, and Methane in Gas, Determination of, by Combustion in a Quartz Tube, 341
——— in Humus, Methods of Determining, Contribution to, 163
Nitrogenous Constituents of Hops, 459
——— of Raw Caoutchouc, the Estimation of, Method for, 272
——— Substances in Barley and Malt. III. Transformation of the Proteins during Mashing, Boiling of Wort, and Fermentation, 496
———, Soluble, Estimation of, as a Criterion of the Quality of Flour, 148
II.—Nitroglycerine and Cordite. I.—Guncotton. III.—Theoretical. Heat Test, 275
Nitroglycerine, Behaviour of, when Heated, 115
———, Quantitative Separation of Nitrosubstitution Compounds from, 513
p-Nitrophenylhydrazine, Detection of Formaldehyde with, 466
Nitroprusside Reaction of Urine, 154
α-nitroso-β-naphthol, Precipitation of Palladium by Means of, 289
Nitrosubstitution Compounds, Quantitative Separation of, from Nitroglycerine, 513
Nitrous Acid and Formaldehyde, Interaction between, 67
——— and Thiocarbamide, in Presence of Acids of Different Strength, Interaction of, and Estimation of Nitrites by Means of Thiocarbamide, 440
——— in the Presence of Ferric Salts, Detection of, 289
——— of Nitric Acid, Detection of, 585
———, Reduction of Manganese Dioxide by, and the Use of this Reaction in the Estimation of Phosphorus in Iron and Steel, without Separation of Silicon, 77
——— Anhydride, the Formation of, Gasometric Estimation of Nitric Oxide and Oxygen, Depending on, 391
Non-Alcoholic Wines of Commerce, So-called, 565
—— Inflammable Cellulose Esters, Analysis and Examination of. Acetylcelluloses, 577
—— Nitrogenous Extractive Matters in Fodder and Foodstuffs, 560
—— Poisonous and Poisonous Saponins, Detection and Differentiation of, by Hæmolysis, 151
Nuts, Cohune, Oil of, 433
——, Hickory, and Hickory Nut Oil, 498
——, Manketti, Oil of, 105

OBITUARY NOTICES:
  Adams, Dr., M.A., F.R.C.S., F.I.C., 241, 305
  Claudet, A. C., 53
  Heron, John, 185
  Lewkowitsch, Julius, 493
  Mosenthal, H. de, 2
Oil (Arachis), Earthnut, in Olive Oil, Detection of, by the Franz-Adler Method, 58
—, Arachis, Composition of, 514
—, Baobab, 498
—, Cocoanut and Palm-Kernel, in Mixtures, the Proportionate Determination of, 255
—, ————, the Composition of, and Alcoholysis, 8
—, ————, the Quantity of, Present in a Butter, Calculation of, from the Polenske Value of the Mixture, 558
—, Cottonseed, Modification of Halphen's Test for, 68
—, Earthnut, Nitrobenzene in, Estimation of, 457
—, Essential, of Cocoa, 20
—, ————, of Jamaica Ginger, 56
—, from the Seeds of *Ximenia Americana*, 147
—, Fusel, Valuation of, 66
—, Hickory Nut, and Hickory Nuts, 498
— in Grains, the Determination of, Rapid Control Method for, 57
—, Kapok, and Kapok Seeds, 467
—, Kaya, Fatty Acids of, 458
—, Linseed, Adulteration in, the Detection of, 3, 104
—, ————, Effect of Certain Pigments on. Manganese Content of Raw Linseed Oil, 215
—, ————, Oxygen Absorption Test for, 159
—, Marine Animal (Thran), in Other Oils and Fats, Detection of, 34
— Mixtures, Fish and Vegetable, Properties of. Studies on Fish Oils, 70
— of Bitter Almonds, and Benzaldehyde, Estimation of, 19
— of Cohune Nuts, 433
— of Manketti Nuts, 105
— of Turpentine, Maumené Figure of, 338
————————, Petroleum Products in, Detection of, by Means of the Maumené Test, 70
—, Olive, Earthnut (Arachis) Oil in, Detection of, by the Franz-Adler Method, 58
—, ————, Rape Oil in, Detection of, 434
—, Palm-Kernel and Cocoanut, in Mixtures, the Proportionate Determination of, 255
—, Pine Nut, 275
(—, Rape, Detection of). Fractional Precipitation of Fatty Acid Mixtures, 114
—, ————, in Olive Oil, Detection of, 434
—, ————, in Raw Linseed Oils, the Estimation of, and Hexabromide Number of Fatty Oils, 158
— Seeds from the Anglo-Egyptian Sudan, 323
—, Soya Bean, Colour Reaction of, 36
—, the "Cold Test" of, Experiments on, 34
—, Tung, Examination of, 116
—, Turpentine, Examination of, by the Methods of the German Pharmacopœiæ, 501
—, Whale, Hardened, Colour Reactions of, 433
Oils and Fats, Other, Marine Animal Oil (Thran) in, Detection of, 35
—, Blown, Rosin in, Estimation of, 469
—, Colour Reactions of, with Certain Phenols in the Presence of Nitric Acid, 433
—, Essential, from Various Countries, 559
—, ————, Glyceryl Acetate in, Estimation of, 148

Oils, Essential, in Absinthe and Similar Liqueurs, and in Alcoholic Solutions of Essential Oils, Estimation of, 418
——, ————, Oxidation Assay of, 69
——, Fatty Oils, Hexabromide Number of, and the Estimation of Rape Oil in Raw Linseed Oils, 158
——, Fish, Studies on. III. Properties of Fish and Vegetable Oil Mixtures, 70
—— from *Manihot Ceara* and *Funtumia Elastica*, Examination of, and a Comparison of their Properties with those of Linseed and *Hevea* Oils, 259
——, Hardened, Analysis of, 432
——, "Hardened," Note on, 102
——, Lemon Grass, from India, 160
——, Lubricating, Viscosity of, 36
——, Marine Animal, Hardened, 373
——, ————, Lubricating, and Transformer, Evaporation Test for, 333
——, Mineral, in Oleins of Wool Fat, Detection of, 35
——, Raw Linseed, the Estimation of Rape Oil in, and Hexabromide Number of Fatty Oils, 158
——, —— Paint, Drying Rates of, 468
——, Solidified (Hydrogenised), and the Detection of Arachidic Acid, 160
——, Steam Turbine, Examination of, 468
——, Tar, and Crude Carbolic Acid, Phenol in, Estimation of, 162
——, Turpentine, from India, 165
——, Vegetable, in Margarine, Detection of, 21
"——, Wood," from China and Japan, 580
Oleic and Stearic Acids, Mixtures of, Solidifying and Melting Points of, 579
Oleins of Wool Fat, Mineral Oils in, Detection of, 35
Opacity and Hiding Power of Pigments, Tests on, 161
Opium and Opium Preparations, Morphine in, Estimation of, 150
Ores and Minerals, Valuation of, Notes on, and on Metallurgical Calculations, 171
—— Containing Arsenic, Antimony, or Bismuth, Copper in, Electrolytic Estimation of, 222
——, Copper in, Thiocyanate-Permanganate Method for, 223
——, Rocks, Slags, and Spiegels, Manganese in, Volumetric Determination of, 170
——, Zinc, Fluorine in, Estimation of, 475
——, —— in, Determination of, 298
Organic Acids, Determination of Carboxyl Groups in, Improved Apparatus for, 299
ORGANIC ANALYSIS, ABSTACTS, 31, 65, 111, 154, 211, 271, 329, 371, 424, 464, 508, 574
Organic Compounds, Oxygen in, Direct Estimation of, 216
————————, Some, the Estimation of, Permanganate (Alkaline) in, 215
———— Matter in Clays, Estimation of, 374
————————, Method of Standardising Disinfectants with or without, 422
——, Phenol in Presence of, Estimation of, 336
—— Phosphorus Compounds, Hydrolysis of, by Dilute Acid and Dilute Alkali, 278
—— Solvents, Evaporated, Apparatus for Recovery of, 398
—— Substances, Iodine in, Estimation of, 427
————————, Moisture in, Determination of, 177
————————, Other, and Foods, Moisture in, Determination of, 177
Small Quantities of Mercury in, Estimation of, 515
———— ————————, Sulphur in, Estimation of, 515
Osmium Tetroxide as an Oxygen Carrier and the Rendering Active of Chlorate Solutions, 78
Oxalate, Precipitation as, and Titration with Permanganate, Estimation of Lead, Nickel, and Zinc by, 122
Oxidation Assay of Essential Oils, 69

Oxidation of Fatty Acids and Unsaponifiable Matter during Process of Fat Estimation by the Kumagawa-Suto Method, 432
——— Products of Cholesterol, Study of the, 209
——— with Bromine, Identification of Small Amounts of Dyestuffs by, 376
Oxide, Nitric, and Oxygen, Gasometric Estimation of, Depending on the Formation of Nitrous Anhydride, 391
———, ———, Estimation of, 171
———, ———, the Estimation of, New Gasometric Method for (I.), 45
——— of Iron, Hydrated, Valuation of, by Means of the "Sulphuretted Hydrogen Burette," 522
Oxidising Agents, an Electro-Chemical Indicator for, 353
——— Power, Estimation of, and Application of Dialysis as Aids in Judging Soils, 369
Oxygallol Derivatives, Colour Reactions of Alkaline Earths with, 473
Oxygen Absorption Test for Linseed Oil, 159
———, Action of Tartaric Acid on Tin in the Presence of, 472
———, Active, in Perborates and in Washing Powders Containing Perborates, 441
——— and Nitric Oxide, Gasometric Estimation of, Depending on the Formation of Nitrous Anhydride, 391
———, Available, in Per-Salts, Estimation of, by Means of Titanium Trichloride, 78
——— -Carrier, Osmium Tetroxide as an, and the Rendering Active of Chlorate Solutions, 78
———, Dissolved, in Water, the Determination of, Apparatus for, 396
———, ———, ———, ———, Practical Field Method for, 397
———, ———, ———, the Estimation of, Use of Sodium Potassium Tartrate in, 442
———, Elementary, New Delicate Reaction for, 172
———, in Copper and Brass, Determination of, 520
——— in Iron and Steel, Determination of, Application of Electric Resistance Furnace to, 168
——— in Organic Compounds, Direct Estimation of, 216
———, Moist, Combustion with, Estimation of Carbon by, 271
———, the Absorption of, by Sewage Effluents, Measurement of, 123
——— under Pressure, Estimation of Total Carbon in Steel and Ferro-Alloys by Combustion in, 285
Oxymethylanthraquinones, Extracts of Drugs Containing, Detection of Aloes in, 321
Ozone, and Hydrogen Peroxide, Estimation of, 344

Paint Oils, Raw, Drying Rates of, 468
Paints and Especially Lithopones, Analysis of, 334
Palladium, Estimation of, and its Separation from Copper and Iron, 79
———, Precipitation of, by means of α-nitroso-β-naphthol, 289
———, Separation of, from Gold, Platinum, Rhodium, and Iridium, 524
Palmitate, Potassium, Use of, in Water Analysis, 127
Palmitic and Stearic Acids Present in Lard, Mixed Glycerides of, 204
β-Palmitodistearin and α-Distearin, the Synthetic Preparation of, and Nomenclature of Mixed Glycerides, 214
Palm-Kernel and Cocoanut Oil in Mixtures, the Proportionate Determination of, 255
Paper, Mechanical Wood in, Simple Reagent for the Detection of, 434
———, Neutral Red, as Indicator for Alkalimetry of Serum, 328
———, Rosin in, Quantitative Determination of, 516
———, the Determination of Starch in, Polarimetric Method for, 380
Papers, Parchment, Testing, 161
Paraffin Wax and Ceresin in Their Mixtures, Estimation of, 37

Paraffins in Lanolin, Detection of, 458
Pastry Containing Eggs, Analysis of, 417
Pathogenic Microbes in "Raw" River Water and in Crude Sewage, Search for, 370
Pentoses and Methylpentoses in Grapes and Wines, Estimation of; Investigations on Lead Precipitates from Wines, 25
———— in Diabetic Urine, Detection of, 64
———— in Presence of Other Sugars, Estimation of, by Means of the Spectroscope, 333
Pepper, Red, Different Varieties of, Composition of, 418
Peppercorns, Adulterated, 58
Pepsin Activity, Determination of, 210
——— and Peptase, Distinctions between, 507
———, the Activity of, and the Acidity-Maximum of Peptic Digestion, Mett's Method for Determining, 29
Peptase and Pepsin, Distinctions between, 507
Perborates, and Washing-Powders Containing Perborates, Active Oxygen in, Estimation of, 441
Perchlorate in Potash Fertilisers, Estimation of Potassium as, 124
Periodate in Presence of Persulphate, Estimation of, and Action of Persulphate on Iodate, 479
Permanganate [Alkaline] in the Estimation of Some Organic Compounds, 215
Permanganate, Titration with, and Precipitation of Oxalate, Estimation of Lead, Nickel, and Zinc by, 122
Permeability of Cells by Alkalis, the Estimation of, Indicator Method for, 327
————— of Soils to Water, Determination of, 49
Peroxide, Hydrogen, and Ozone, Estimation of, 344
————, Lead, in Minium, Rapid Method of Estimating, 343
Peroxydase Reaction of Milk (Influence of Preservatives, etc.), 325
Per-Salts, Available Oxygen in, Estimation of, by Means of Titanium Trichloride, 78
Persulphate, Action of, on Iodate, and Estimation of Periodate in Presence of Persulphate, 479
Persulphuric Acid, Iodimetric Estimation of, 293
Peru Balsam, Analysis of, 150
—————, Reaction of, 58
Petermann's Ammonium Citrate Solution, Preparation of, 338
Petroleum and its Products, Water in, Estimation of, 165
————, California, Fractionation of, by Diffusion through Fuller's Earth, 469
————, Crude, from the Gold Coast, 162
———— or Asphalt Products in Tars, Small Amounts of, Application of the Dimethyl Sulphate Test for Determining, 277
———— Pitch and Natural Asphaltum, in Residues from the Distillation of Coal-Tar, Detection of, 157
———— Products, Determination of Sulphur in Fuels, Especially, 38
———————— in Oil of Turpentine, Detection of, by Means of the Maumené Test, 70
———————, Specifications for, Preparation of, 335
———— Spirit in Turpentine, Estimation of, 464
———— Testing, Methods and Apparatus Used in. Part II.: Viscosimetry, 397
Pettersson-Palmqvist Apparatus, Portable, 178
Pharmacopœia, the German, Examination of Turpentine Oil by the Methods of, 501
Phenol and Salicylic Acid, Benzoic Acid in the Presence of, Detection of, 371
——— Disulphonic Acid Method, Determination of Nitrates by, Influence of Chlorine on, 339
———, Hydriodic Acid in Presence of, Methoxyl Estimations with, 33
——— in Crude Carbolic Acid and Tar Oils, Estimation of, 162

Phenol in Presence of Organic Matter, Estimation of, 336
——, the Bromination of, for Quantitative Determinations, Effect of Temperature, Acid Concentration, and Time in, 335
Phenols, Certain, Colour Reactions of Oils with, in the Presence of Nitric Acid, 433
—— in Creolins, Estimation of, 325
Phosphate, Zinc, and Zinc Ammonium Phosphate, Estimation of Zinc as, 443
Phosphates, Water-Soluble, in Ammonium Superphosphates, Estimation of, 392
Phosphoric Acid, Estimation of, 344
————, Quantitative Volatilisation and Separation of, from the Metals of the Copper Group, and from Aluminium, Tin, and the Alkali Metals, 525
————, Separation of, by Means of Stannic Acid in Qualitative Analysis, 292
———— Soluble in Citric Acid in Thomas Slag, Estimation of, 171, 526
———— and Boric Acids, Titration of, 124
Phosphorus Compounds, Organic, Hydrolysis of, by Dilute Acid and Dilute Alkali, 278
——— in Commercial Acetylene, Determination of, 46
——— in Feeding-Stuffs and Manures, Estimation of, 563
——— in Ferrotungsten, Metallic Tungsten Powder, Tungsten Oxide, and Tungstic Acid, Determination of, by Direct Solution, 290
——— in Iron and Steel, Estimation of, 291
——— in Iron and Steel, the Estimation of, without Separation of Silicon, Reduction of Manganese Dioxide by Nitrous Acid, and the Use of this Reaction in, 77
——— in Lecithin, Estimation of, 58
——— in Steel, Determination of, Rapid Method for, 442
——————, Volumetric Estimation of, by Macagno's Method, 526
——— in Steels Containing Vanadium, Determination of, 478
——— in Vanadium Steel and Ferrovanadium, Determination of, 587
——— Sesquisulphide, White Phosphorus in, Detection and Estimation of, 80
———, White, in Match Compositions, Detection of, 289
Physico-Chemical Behaviour of Human Milk, Cow's Milk, and Buttermilk, Effect of Boiling on, 205
———— Estimation of Sulphates, Tartaric Acid, Calcium, and Magnesium in Wine, 421
———— Volumetric Method, New, 294
Physiological Standardisation of Digitalis Preparations, 462
Phytic Acid in Cottonseed Meal and Wheat Bran, 419
Phytin, Investigation of, 279
Picric Acid, Estimation of Quinine by Titration with, 105
———, the Precipitation of Albumin by, Studies of the Conditions of, 329
Pigments, Certain, Effect of, on Linseed Oil. Manganese Content of Raw Linseed Oil, 215
———, the Opacity and Hiding Power of, Tests on, 161
Pine-Nut Oil, 275
Pipette, Automatic, 534
Pitch, Petroleum, and Natural Asphaltum in Residues from the Distillation of Coal Tar, Detection of, 157
Plant Ashes, Fertilisers, and Soil Extracts, Potassium in, Estimation of, 47
—— Extracts, Methods of Estimation of Carbohydrates, especially in, 504
——, the Rice, Sulphur and Chlorine in, Estimation of, 572
——, the Tobacco, Enzymes of, 506
Plants, Green, Respiratory Quotients of, 152
———, Hydrocyanic Acid in, the Determination of, with Special Reference to Kaffir Corn, Colorimetric Method for, 569

Plants, the Juices of, Occurrence of Formaldehyde in, 152
Plated Articles, Nickel in, Detection of, 45
Platinum and Gold, Detection of, 476
——— Electrodes, Light, Electro-Analysis with, 43
Estimation of, 294
–, Estimation of, by Precipitation as Sulphide, 173
–, Gold, Rhodium, and Iridium, Separation of Palladium from, 524
Poisonous and Non-Poisonous Saponins, Detection and Differentiation of, by Hæmolysis, 151
Polarimetric Determination, Direct, of Sucrose in Presence of Certain Reducing Sugars, Method for, 71
Estimation of Lactose, Influence of Ammonium Sulphate on, 217
— Method for the Determination of Starch in Paper, 380
Polenske "Difference Value" for the Detection of Certain Animal Fats and its Theoretical Basis, 204
——— Value of the Mixture, Calculation of the Quantity of Cocoanut Oil Present in a Butter from, 558
Polythionate in Presence of Thiosulphate and Free Sulphurous Acid, Estimation of, 392
Port Wine, Composition of, 368
Portland Cement, Examination of, 437
Potash Fertilisers, Potassium as Perchlorate in, Estimation of, 124
Potassium and Sodium when Together in Solution, Estimation of the Carbonates and Hydroxides of, 166
——— as Perchlorate in Potash Fertilisers, Estimation of, 124
——— Bichromate in Milk, Detection of, 563
——— Bromate, Volumetric Method for the Estimation of Tin by Means of, 229
——— Chloride in Hydrofluoric Acid Solution, Separation of Niobium and Tantalum by Means of, 226
———, Detection of, by Means of Tartaric Acid, 295
——— Dichromate, Alteration of Milk Treated with, 325
———, Estimation of, by Cobaltinitrite Method, 81, 294
——— Fluoride, Alcohols, and Water, Systems Containing, Equilibria in. Method for Estimating Water in Alcohol, 425
——— Hydroxide, Estimation of Sodium Carbonate and Copper in Mixtures of Soda Ash and Blue Vitriol by Titration with, 340
——— in Fertilisers, Soil Extracts, and Plant Ashes, Estimation of, 47
Palmitate in Water Analysis, Use of, 127
Powder, Black, and Dynamite, Analysis of, 514
———, ——— Mustard, 500
Powders, Mineral, the Specific Gravity of, Determination of, 296
———, Washing, Containing Perborates, and Perborates, Active Oxygen in, Estimation of, 441
Precipitates, Small, Quantitative Manipulation of, 302
Precipitation Method, the Serum, Detection of Adulteration by, 152
Precipitin Reactions, Application of, to Seed Identification, 210
(Preservatives, etc., Influence of). Peroxydase Reaction of Milk, 325
Pressure, Combustion in Oxygen under, Estimation of Total Carbon in Steel and Ferro-Alloys by, 285
———, Diminished, the Continuous Evaporation of Large Volumes of Liquid under, Apparatus for, 591
Propane, Methane, and Ethane, Mixtures of Hydrogen with, Analysis of, 121
———, ———, Ethane, and Isobutane, Mixtures of Hydrogen with, Analysis of, 276

Protein Hydrolysis, Tryptic, Measurement of, by Determination of the Tyrosine liberated, 153
——— Precipitating Reagent in the Estimation of Lactose in Milk, Aluminium Hydroxide as a, 418
Proteins, Colour Reaction of, 380
——— in Milk, Estimation of, by Aldehyde Titration, 266
——— (Kidney, Liver, and Cancer Proteins), Differentiation of: Experiments on Antigen and Antibody Reactions, 26
———, Milk, Solubility of, in the Constituents of the Serum, 570
———, Nephelometric Determination of: Casein, Globulin, and Albumin in Milk, 571
———, Some, Density and Solution Volume of, 279
———, the Estimation of *l*-Tyrosine in, Accuracy of, 219
———, Transformation of, during Mashing, Boiling of Wort, and Fermentation. Nitrogenous Substances in Barley and Malt, 496
Proteolysed Substances, Blood, and Urine, the Estimation of α- and β-Amino Acids and Certain Derivatives in, Micro-chemical Method for, 567
Protoplasmalipoids, Especially in Muscular Tissue, Microscopical Detection of, 423
Pulp, Sulphite, Bleaching Quality of, New Technical Determination of, 72
Purine Bases and Uric Acid, Precipitation of, by Zinc Salts, 424
——— Bases in Blood, Estimation of, 107
——— ———, Precipitation of, from Meat Extract and Urine by Means of Zinc Salts, 572
Pyknometer, Improved, for Density of Solids, 130
Pyridine Bases, Directions for Testing, 471
Pyrites and Other Sulphides, Sulphur in, Estimation of, 480
———, Arsenic in, Estimation of, 117
———, Burned, Sulphur in, Rapid Estimation of, 529
———, Burnt, Copper in, Rapid Estimation of, 389

Quantitative Dialysis, New Apparatus for, 232
——— Manipulation of Small Precipitates, 302
——— Separations, Some, of Neodymium, 343
——— Volatilisation and Separation of Phosphoric Acid from the Metals of the Copper Group, and from Aluminium, Tin, and the Alkali Metals, 525
Quartz Tube, Combustion in, Determination of Hydrogen, Nitrogen, and Methane in Gas by, 341
Quercitin in Wine, Estimation of, 267
Quininæ et Ferri Citras and *Extractum Ferri Pomatum* Direct Iron Estimation in, 562
Quinine Assay, 59
———, Estimation of, by Titration with Picric Acid, 105
Radio-Activity, Measurement of, Estimation of Uranium by, and Radiation of Uranium Solutions, 345
——— of Solutions of Uranium Salts: Method for the Estimation of Uranium, 231
Radium Standard, International, Purity of the, 173
(Railway Sleepers) Treated Ties, Zinc in, Estimation of, 589
Raisin Wines, Sweet, Glycerol in, Estimation of, 24
(Rape Oil, Detection of) Fractional Precipitation of Fatty Acid Mixtures, 114
——— in Olive Oil, Detection of, 434
Rays, Ultra-Violet, the Absorption of, by the Fatty Acids and their Isomeric Esters, Quantitative Study of, 113

Reaction-Velocity in a Heterogeneous System at High Pressures, Bomb with Stirring Gear for the Measurement of. New High-Pressure Reducing Valve, 590
———, Bettendorf's, Detection of Arsenic by Means of, 221
Reagent, Lloyd's, Precipitation of Alkaloids by, 426
———, New, for Bromine and Chlorine, 119
———, ———, for the Detection of Traces of Blood, 422
———, Simple, for the Detection of Mechanical Wood in Paper, 434
Red Pepper, Different Varieties of, Composition of, 418
Reducing Sugars, Certain, the Direct Polarimetric Determination of Sucrose in Presence of, Method for, 71
——— ———, Estimation of, by Lehmann's Method, 219
Redwood's and Engler's Viscosimeters, Comparison of, 398
Refractometer, the Immersion, Use of, in Examining American Beers Made from Malt and Unmalted Cereals, 456
——— ——— Value of the Serum, Detection of Added Water in Milk by Means of the, 500
Reichert-Meissl Value of Fats, Influence of Antiseptic Agents on the, 68
Report, Final (July 25, 1912), International Commission for Unification of the Testing Methods of the Stability of Explosives, 377
———, Law. Demerara Sugar, 595
——— of the Acting Chemist for 1912, U.S. Department of Agriculture, 131
——— of the Connecticut Agricultural Experiment Station for the Year 1912, 541
——— of the Government Chemist upon the Work of the Government Laboratory for the Year Ended March 31, 1913, with Appendices, 536
on the Present State of Rubber Analysis, 336
——— on the Work of Inspectors of Foods for the Year 1911-1912, 131
Report, Preliminary, of the Committee on Coal Analysis, of the American Society for Testing Materials and the American Chemical Society, 374
———, Seventh Annual, March 31, 1913: Metropolitan Water Board, 487
——— to the Local Government Board on Bacterial Food-Poisoning and Food Infections, 535
———, Western Australia. The Health Act, 1911: Food Standards and Regulations, 179
Residual Reduction of Blood, 327
Residuals, Native Bitumens and their, Characteristics and Differentiation of, 371
Residues from the Distillation of Coal-Tar, Natural Asphaltum and Petroleum Pitch in, Detection of, 157
———, the Recovery of Iodine from, 409
Resins and Substitutes in Caoutchouc Articles, the Estimation of, Use of the Köttstorfer Value for, 337
——— and Waxes of Kapok and Akon Fibres, 580
———, Caoutchouc, Studies on, 465
Respiratory Quotients, Determination of, 108
——— ——— of Green Plants, 152
Rhodium, Gold, Platinum, and Iridium, Separation of Palladium from, 524
Rice Plant, Sulphur and Chlorine in the, Estimation of, 572
River Water, "Raw," and Crude Sewage, Pathogenic Microbes in, Search for, 370
Rocks and Minerals, Silicate, Estimation of Water in, 47
Rocks and Minerals, Small Quantities of Manganese and Chromium in, Estimation of, 226
———, Slags, Ores, and Spiegels, Manganese in, Volumetric Determination of, 170

Roselle, 60
Rosin in Blown Oils, Estimation of, 469
—— in Paper, Quantitative Determination of, 516
—— in Soap, Estimation of, 280
Rotatory Power at Constant Temperature, Electrically Heated Apparatus for Measuring, 300
Rubber Analysis, the Present State of, Report on, 336
——, Crude, Caoutchouc in, Estimation of, 511
——, Direct Determination of, 337
—— Solutions, the Viscosity of, Estimation of, 272
——, the Preparation of, for Analysis, 550
——, the Ultimate Estimation of "Sublimed White Lead" in, and Determination of Lead Sulphate, 44
—— Wares, Particularly Insulated Wires, Estimation of Caoutchouc in, 111
Rubidium and Cæsium, Salts of, Behaviour of, towards Salts of Heavy Metals, 283
Rum, Jamaica, Method of Distinguishing, from Artificial Rum, 368
Rums, Composition of, 500
Russian Honey, Composition of, 149

Saccharin in Foods, Estimation of, 326
—— in Vermouth, Detection of, 22
Saffron Containing Formaldehyde, 420
—— in Confectionery, Detection of, 206
Salicylic Acid and Phenol, Benzoic Acid in the Presence of, Detection of, 371
—— in Fruit Juices, Detection and Estimation of, 420
"Salt Error" in Colorimetric Estimation of Hydrogen Ion Concentration of Sea-Water, 394
Salts, Calcium and Magnesium, Behaviour of, with Soap Solutions, and the Determination of Hardness of Water, 436
——, Ferric, Nitrous Acid in the Presence of, Detection of, 289
——, Gold, Delicate Colour Reactions of, 54
——, Halogen, Bromine in, Iodic Acid Method of Estimating, 166
——, ——, the Estimation of Bromine in, Preparation of Selenic Acid and Sodium Selenate as Reagents for, 295
——, Magnesium and Calcium, Behaviour of, with Soap Solutions, and the Determination of Hardness of Water, 436
—— of Cæsium and Rubidium, Behaviour of, towards Salts of Heavy Metals, 283
——, Uranium, Radio-Activity of Solutions of: Method for the Estimation of Uranium, 231
——, Zinc, Precipitation of Purine Bases from Meat Extract and Urine by Means of, 572
——, ——, —— of Uric Acid and Purine Bases by, 424
Samna and Butter, Egyptian, 242
——, Note on, 252
Samples, Milk, Preservation of, for Analysis, 325
Sampling Coal Deliveries, 330
Sand, Monazite, Separation of Thorium from, by Means of Hypophosphorous Acid. Detection of Cerium, 443
Saponin, Detection of, 151
——, the Estimation of, Methods for, 22
Saponins, Poisonous and Non-Poisonous, Detection and Differentiation of, by Hæmolysis, 151

Schumacher-Jung and Farup Methods for the Estimation of Mercury in Urine, Comparison of, 111
Sections of Crystalline Substances, Preparation of, 483
——— of Fractures of Steel for Microscopic Examination, Preparing, 528
Seed, "Cheyi," 323
—— Identification, Application of Precipitin Reactions to, 210
—— -Testing, Biological Considerations in Relation to, 210
Seeds, Kapok, and Kapok Oil, 467
——— of *Cicer Arietinum*, L., (Chick Pea), Composition of, 496
——— of *Ximenia Americana*, Oil from, 147
———, Oil, from the Anglo-Egyptian Sudan, 323
———, the Vitality of, Method for Detecting, 370
Selenic Acid and Sodium Selenate as Reagents for the Estimation of Bromine in Halogen Salts, Preparation of, 295
——————, Application of, to the Estimation of Bromide in Presence of Chloride, 227
Semen, Gold Bromide Test for, 29
Sensitising Mylius's Reaction for the Detection of Cholalic Acid and Other Biliary Acids, Method of, 506
Sensitive Reaction for Molybdic Acid, 43
———————— of Molybdenum, 478
Separating Funnel, New, 234
Serum and Blood-Corpuscles, Chlorine Content of Blood and its Distribution between, 327
———, Blood-, Lipoids in, Estimation of, 28
———, Constituents of the, Solubility of Milk Proteins in, 570
———, Neutral Red Paper as Indicator for Alkalimetry of, 328
——— Precipitation Method, Detection of Adulteration by the, 152
———, the Refractometer Value of, Detection of Added Water in Milk by Means of, 500
Sewage, Crude, and "Raw" River Water, Pathogenic Microbes in, Search for, 370
——— Disposal, Royal Commission on: Appendix to Eighth Report, March, 1913, 539
——— Effluents, the Absorption of Oxygen by, Measurement of, 123
Shellac Analysis and the Detection of Small Amounts of Colophony in Shellac, 38
———, Bleached, Water in, Estimation of, 280
Silica, Thorium, Lanthanum, Cerium, Erbium, and Didymium, Separation of Tungsten from, 481
Silicate Minerals and Rocks, Water in, Estimation of, 47
Silicates, Opening up, 124
Silicon, Separation of, Reduction of Manganese Dioxide by Nitrous Acid, and the Use of this Reaction in the Estimation of Phosphorus in Iron and Steel without, 77
Silver Determination, Gay-Lussac Method of, 228
——— Precipitation, Titration of Uric Acid in Urine after, 329
Skatol and Indol Together in Fæces, Estimation of, 30
Skita's Hydrogenising Apparatus, Simple Modification of, 484
Slag, Thomas, Phosphoric Acid Soluble in Citric Acid in, Estimation of, 171, 526
Slags, Rocks, Ores, and Spiegels, Manganese in, Volumetric Determination of, 170
Smell, the Sense of, Use of, in Volumetric Estimations, 594
Soap, Rosin in, Estimation of, 280
——, Sodium Borate in, Determination of, 461
—— Solutions, Behaviour of Calcium and Magnesium Salts with, and the Determination of Hardness of Water, 436
Soaps, Fatty Acids in, Estimation of, Rapid Volumetric Method for, 432
——— in Lubricating Greases, Estimation of, 162

SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS, PROCEEDINGS OF, 2, 54, 87, 135, 186, 241, 307, 353, 407, 447, 496, 549
Soda Ash and Blue Vitriol, Mixtures of, Estimation of Sodium Carbonate and Copper in, by Titration with Potassium Hydroxide, 340
Sodium and Potassium when Together in Solution, the Carbonates and Hydroxides of, Estimation of, 166
—— Borate in Soap, Determination of, 461
—— Carbonate and Copper in Mixtures of Soda Ash and Blue Vitriol, Estimation of, by Titration with Potassium Hydroxide, 340
—— Hypobromite, Estimation of Urea in Urine by, 269
—— Hypophosphite, Estimation of Copper by Means of, 75, 519, 520
—— Iodide, Purification of Acetone by Means of, 424
—— Potassium Tartrate, Use of, in the Estimation of Dissolved Oxygen in Water, 442
—— Selenate and Selenic Acid, as Reagents for the Estimation of Bromine in Halogen Salts, Preparation of, 295
Soil Analysis, the Lime-Magnesia Ratio in, Significance of, 126
—, Biochemical Examination of, 462
— Extracts, Fertilisers and Plant Ashes, Potassium in, Estimation of, 47
—, Manganese in, Estimation of, 343
—, the Estimation of the Reaction and Basicity of, Significance of, 462
Soils, Absorbed Bases in, Estimation of, 424
——, Aids in Judging, Application of Dialysis and Estimation of Oxidising Power as, 369
——, Carbonates in, Some Qualitative and Quantitative Methods for, Comparison of, 163
——, Mineralogical Analysis of, 125
——, the Permeability of, to Water, Determination of, 49
Solidified (Hydrogenised) Oils and the Detection of Arachidic Acid, 160
Solidifying and Melting Points of Mixtures of Stearic and Oleic Acids, 579
Solids and Liquids, Extraction Apparatus for, 351
——, Density of, Improved Pyknometer for, 130
——, Finely Divided, Improved Apparatus for Extracting, with Volatile Solvents, 444
——, Total, of Milk, 21
Soluble Nitrogenous Substances, Estimation of, as a Criterion of the Quality of Flour, 148
Solubilities, Estimation of, 84
Solubility of Milk Proteins in the Constituents of the Serum, 570
Solution and Density Volume of Some Proteins, 279
Solvent, Cryoscopic, Ethylene Dibromide as, 32
———, Formic Acid as a, 513
———, Investigation of the. Part I. Analysis of Spirit Varnishes, 164
Solvents, Immiscible, Prevention of Emulsification in Extractions by, 48
———, Organic, Evaporated, Recovery of, Apparatus for, 398
———, Volatile, Improved Apparatus for Extracting Finely Divided Solids with, 444
Soya Bean Oil, Colour Reaction of, 36
Specific Gravities of Milk, Extension of Vieth's Table for Correcting the, 460
——— Gravity Bottle and Dilatometer, Combination, 485
————— of Mineral Powders, Determination of, 296
—————, Unusual Case of, 50
——— Heat Apparatus, New Designs for, 484
Specifications for Petroleum Products, Preparation of, 335
Spectroscope, Estimation of Pentoses in Presence of Other Sugars by Means of the, 333
Spectroscopic Detection of Magnesium, Calcium, Strontium, and Barium, 584

Spiegels, Rocks, Slags, and Ores, Manganese in, Volumetric Determination of, 170
Spirit Varnishes, Analysis of. Part I.: Investigation of the Solvent, 164
Spirits, Higher Alcohols in, Estimation of, 564
——, Methyl Alcohol in, Estimation of, 56
—— of Camphor, the Examination of, Use of Miscibility Curves in, 204
Springs and Filter-Beds, the Testing of, New Rapid Method for the Bacteriological Examination of Water and its Application for, 30
Stability of Explosives, Unification of the Testing Methods of, International Commission for Final Report (July 25, 1912), 377
Standard for Volumetric Analyses, Mercuric Oxide as, 439
——, the International Radium, Purity of, 173
Standardisation of Hydrometers, 395
——, Physiological, of Digitalis Preparations, 462
Standards, International, for Coloured Fluids, and a Suggested Plan for Such Standardisation, 394
Stannic Acid, Separation of Phosphoric Acid by Means of, in Qualitative Analysis, 292
Stannous Chloride and Chlorates, Determination of, by Means of Methylene Blue, 126
Starch Granules, the Gelatinising Temperatures of, Researches on, 74
—— in Commercial Products, Estimation of, 472
—— in Paper, the Determination of, Polarimetric Method for, 380
——, the Estimation of, by Ewer's Process, Apparatus for Use in, 445
Statistics, Wine, for the Years 1911-1912, 566
Steam Turbine Oils, Examination of, 468
Stearic and Oleic Acids, Mixtures of, Solidifying and Melting-Points of, 579
—— and Palmitic Acids Present in Lard, Mixed Glycerides of, 204
Stearin of Wool Fat, 37
Steel and Ferro-Alloys, Total Carbon in, Estimation of, by Combustion in Oxygen under Pressure, 285
—— and Iron, Carbon in, Rapid Method for Estimating, 384
——, Determination of Oxygen in, Application of Electric Resistance Furnace to, 168
——, Phosphorus in, Estimation of, 291
——, the Estimation of Phosphorus in, without Separation of Silicon, Reduction of Manganese Dioxide by Nitrous Acid, and the Use of this Reaction in, 77
——, Titanium in, Colorimetric Method for, 588
——, Cast Iron, and Iron Alloys, Total Carbon in, Rapid Determination of, 442
——, Chromium in, Determination of, Colorimetric Method for, 285
——, Cobalt, and Uranium in, Estimation of, 527
——, Estimation of Vanadium in, by the Hydrogen-Peroxide Colour Method, 589
——, Fractures of, Preparing Sections of, for Microscopic Examination, 528
——, Phosphorus in, Determination of, Rapid Method for, 442
Volumetric Estimation of, by Macagno's Method, 526
Uranium and Cobalt in, Estimation of, 527
-, Vanadium, and Ferrovanadium, Phosphorus in, Determination of, 587
Steels and Castings, Manganese in, Estimation of, by the Method of G. von Knorre, 286
——, Chromium and Vanadium in, Determination of, 81
—— Containing Vanadium, Phosphorus in, Determination of, 478
Stirring Gear for the Measurement of Reaction-Velocity in a Heterogeneous System at High Pressures, Bomb with. New High-Pressure Reducing Valve, 590
Strength, Effective, of High Explosives, the Determination of, Methods for, 378

Strontium, Magnesium, Calcium, and Barium, Spectroscopic Detection of, 584
"Sublimed White Lead" in Rubber, the Ultimate Estimation of, and Determination of Lead Sulphate, 44
Substitutes, Butter Fat and its, Viscosity of, 458
——— and Resins in Caoutchouc Articles, the Estimation of, Use of the Köttstorfer Value for, 337
Succinic Acid in Meat Extract and Fresh Meat, Occurrence of, 508
———, Tartaric, and Malic Acids in Wine, Estimation of, 502
Sucrose in Cane Molasses, Determination of, by the Double Polarisation Method, Using Invertase and Acid as Hydrolysts, 281
——— in the Presence of Other Sugars, Estimation of, 71
———, the Direct Polarimetric Determination of, in Presence of Certain Reducing Sugars, Method for, 71
———, the Double Polarisation Method of Determining, Use of Sulphurous Acid in Making the Acid Reading in, 282
Suction Gas Fuels, Testing for Liability to Clog Gas-Engine Valves, Rapid Method of, 332
Sudan Dura, 323
———, the Anglo-Egyptian, Oil Seeds from, 323
Sugar, Cane and Beet, Factory and Refinery Products, Estimation of Traces of Iron in, 59
———, Demerara. Law Report, 595
——— (Dextrose), in Urine, Estimation of, 65
——— Estimation (I.), Bang's Method of, 435
——— in the Blood of Frog or Tortoise, the Estimation of, Sources of Error in, 463
———, Invert [Commercial], Detection of, by Means of β-Naphthol, 217
Sugar, Method of Estimating, 218
Sugars, Certain, Behaviour of, with Diphenylamine and Hydrochloric Acid, 435
———, ——— Reducing, Sucrose in Presence of, Method for the Direct Polarimetric Determination of, 71
———, Estimation of, by the Kjeldahl-Bertrand Method, 218
———, Other, Pentoses in Presence of, Estimation of, by Means of the Spectroscope, 333
Sucrose in the Presence of, Estimation of, 71
———, Reducing, Estimation of, by Lehmann's Method, 219
Sulphate, Aluminium, Estimation of Free Acid and Base in, 283
———, in Wines, Estimation of, 369
———, Barium, Natural and Artificial Differentiation of, 517
——— in Ammonium Sulphate Solution, Estimation of, with Special Reference to the Testing of Illuminating Gas, 382
———, Lead, Determination of, and the Ultimate Estimation of, "Sublimed White Lead" in Rubber, 44
———, Purification of the Precipitate Obtained in Estimating Barium as, 339
Sulphates, Tartaric Acid, Calcium, and Magnesium in Wine, Physico-Chemical Estimation of, 421
Sulphide, Estimation of Copper as, 582
———, ——— of Platinum by Precipitation as, 173
———, Lead, and Lead Chromate, Solubility of, 476
Sulphides, Alkali, Estimation of, 393
———, Other, and Pyrites, Sulphur in, Estimation of, 480
Sulphite Pulp, Bleaching Quality of, New Technical Determination of, 72
Sulphites and Sulphurous Acid, Titration of, in Presence of Thiosulphates, New Process for, 297

Sulphites, Thiosulphate in Presence of, Estimation of, 481
Sulphur and Chlorine in the Rice Plant, Estimation of, 572
——— and Nitrogen, Water Formed on Burning Substances Containing, Estimation of, in a Calorimetric Bomb. 347
——— Determinations, Simple Gasoline Gas Generator for, 592
———, Free, in Leather, Estimation of, 467
——— in Burned Pyrites, Rapid Estimation of, 529
——— in Fuels, Especially Petroleum Products, Determination of, 38
——— in Illuminating Gas, the Estimation of, Volumetric Method for, 228
——— in Organic Substances, Estimation of, 515
——— in Pyrites and Other Sulphides, Estimation of, 480
"Sulphuretted Hydrogen Burette," Valuation of Hydrated Oxide of Iron by Means of the, 522
Sulphuric Acid, Free, in Cotton, Detection of, 510
——— Anhydride, Nitric Acid in, Qualitative Detection of, 78
Sulphurous Acid and Sulphites, Titration of, in Presence of Thiosulphates, New Process for, 297
———, Free, and Thiosulphate, Polythionate in Presence of, Estimation of, 392
——— in Air, Estimation of, 127
———, Use of, in Making the Acid Reading in the Double Polarisation Method of Determining Sucrose, 282
Superphosphates, Ammonium, Water-Soluble Phosphates in, Estimation of, 392
Sweets and Liquorice, Glycyrrhizin in, Estimation of, 367
Systems Containing Alcohols, Water, and Potassium Fluoride, Equilibria in. - Method for Estimating Water in Alcohol, 425

Table, Hydrogen, New, 167
——— of Calculated Dry Substance of Milk by the Fleischmann Formula, 324
Tanned Hides, Various East-Indian, the Analysis of, 363
Tannin and Gallic Acid, Colour Reaction for, 467
——— in Tea, the Determination of, 312
———, the Estimation of, Use of Amalgamated Aluminium in, 516
Tantalum and Niobium, Separation of, by Means of Potassium Chloride in Hydrofluoric Acid Solution, 226
Tantalum Electrodes, Application of, to Electro-Analytical Estimation of Copper and Zinc, 481
Tar Oils and Crude Carbolic Acid, Phenol in, Estimation of, 162
Tars, Small Amounts of Petroleum or Asphalt Products in, Application of the Dimethyl Sulphate Test for Determining, 277
Tartaric Acid, Action of, on Tin in the Presence of Oxygen, 472
———, Detection of Potassium by Means of, 295
———, Sulphates, Calcium, and Magnesium in Wine, Physico-Chemical Estimation of, 421
———, the Determination of, 380
——— and Malic Acids in the Same Solution, Determination of, 381
———, Citric, Lactic, and Malic Acids in Musts, Wines, etc., Estimation of, 504
———, Succinic, and Malic Acids in Wine, Estimation of, 502
(Tartrates, for Commercial) Goldenberg Method, 1898 and 1907, 435
Tea, Cocoa, and Coffee, and their Preparations, Xanthin Bases in, Detection and Estimation of, 26, 60
———, Tannin in, the Determination of, 312
Temperatures, Gelatinising, of Starch Granules, Researches on, 74

Temperatures, High, Melting-Point Apparatus for, 483
Textile Dressings, Gelatin in, Detection of, 332
——— Materials, Mercury in, Estimation of, 63
Thermometry, Notes on, 303
Thermo-Regulator, New, 398
——— -Regulators, Gas, Improvement in, 351
Thiocarbamide, Estimation of Nitrites by Means of, and the Interaction of Nitrous Acid and Thiocarbamide in Presence of Acids of Different Strength, 440
Thiocyanate-Permanganate Method for Copper in Ores, 223
Thiosulphate and Free Sulphurous Acid, Polythionate in Presence of, Estimation of, 392
——— in Presence of Sulphites, Estimation of, 481
sulphates, Titration of Sulphurous Acid and of Sulphites in Presence of, New
rocess for, 297
s Slag, Phosphoric Acid Soluble in Citric Acid in, Estimation of, 171, 526
m, Lanthanum, Cerium, Erbium, Didymium, and Silica, Separation of Tungsten from, 481
--, Separation of, from Monazite Sand by Means of Hypophosphorous Acid.
ection of Cerium, 443
) Marine Animal Oil, in Other Oils and Fats, Detection of, 35
reated (Railway Sleepers), Zinc in, Estimation of, 589
uminium, and the Alkali Metals, Quantitative Volatilisation and Separation
of Phosphoric Acid from the Metals of the Copper Group, and from, 525
tection of, 297
Canned Foods, the Estimation of, Iodine Titration Method for the Estimation
f Tin, Especially in Connection with, 23
he Presence of Oxygen, Action of Tartaric Acid on, 472
d, Antimony, and Copper, Alloys of, Analysis of, 584
paration of, from Tungsten Electrolytically, 479
Direct Volumetric Determination of, 82
Estimation of, by means of Potassium Bromate, Volumetric Method for, 229
Animal, and Blood, Small Quantities of Methyl Alcohol in, Estimation and Detection of, 62
Animal, and Urine, the Estimation of Mercury in, Method for, 153
in Iron and Steel, Colorimetric Method for, 588
—— Presence of Iron, Estimation of, 82, 229
, the Separation of, from Aluminium, Use of "Cupferron" in, 390
Trichloride, Estimation of Available Oxygen in Per-Salts by Means

Plant, Enzymes of the, 506
nd Frog, the Blood of, the Estimation of Sugar in, Sources of Error in,

ical Analysis, Antipyrin in, 97
y, Action of "Activated" Aluminium on Alkaloid Extracts, and its Employ-
n, 61, 268
ner, Mineral, and Lubricating Oils, Evaporation Test for, 333
i-Phenols, New Colour Reactions of, 470
rindene Hydrate (Ninhydrin), Colour Reactions of, 579
mine, in Presence of Ammonia, Quantitative Estimation of, 436
otein Hydrolysis, Measurement of, by Determination of the Tyrosine
l, 153
Examination of, 116
Estimation of Calcium as, 283

Tungsten and Arsenic, Separation of, 117

——— and Molybdenum, Electrolytic Separation of Copper from, 224

——— Oxide, Ferrotungsten, Metallic Tungsten Powder, and Tungstic Acid, Determination of Phosphorus in, by Direct Solution, 290

———, Separation of Tin from, Electrolytically, 479

———, ——— of, from Thorium, Lanthanum, Cerium, Erbium, Didymium, and Silica, 481

Tungstic Acid, Quantitative Precipitation of, by Means of Aromatic Amines, 529

Turbine Oils, Steam, Examination of, 468

Turpentine Oil, Examination of, by the Methods of the German Pharmacopœi 501

———, Oil of, Maumené Figure of, 338

———, ———, Petroleum Products in, Detection of, by Means of the Mau Test, 70

——— Oils from India, 165

———, Petroleum Spirit in, Estimation of, 464

——— Sold as a Drug, 564

Turpentines, Wood-, their Analysis, Refining, and Composition, 39

l Tyrosine in Proteins, the Estimation of, Accuracy of, 219

Ultrafiltration, Simple Apparatus for, 486

Ultra-Violet Rays, the Absorption of, by the Fatty Acids and their Isomeri Quantitative Study of, 113

Unsaponifiable Matter and Fatty Acids, Oxidation of, during Process of Fat tion by the Kumagawa-Suto Method, 432

Uranium and Cobalt in Steel, Estimation of, 527

——— Salts, Solutions of, Radio-Activity of: Method for the Estim Uranium, 231

——— Solutions, Radiation of, and Estimation of Uranium by Measur Radio-Activity, 345

-, Volumetric Estimation of, New Method for, 230

Urea in Urine, Estimation of, by Sodium Hypobromite, 269

Uric Acid and Purine Bases, Precipitation of, by Zinc Salts, 424

——— in Urine, Estimation of, 65

———, the Estimation of, Colorimetric Method for, 154

Urine, Acetone in, Estimation of, 110

———, Albumin in, Detection of, 573

——— and Animal Tissues, the Estimation of Mercury in, Method for, 153

——— and Blood, Arsenic in, Estimation of, 106

——— and Blood, Chlorides in, Estimation of, 270

——— and Meat Extract, Precipitation of Purine Bases from, by Mean Salts, 572

———, Diabetic, Detection of Pentoses in, 64

———, Glucose in, Estimation of Small Amounts of, 463

———, Lactic Acid in, Estimation of, 328, 370

———, Mercury in, the Estimation of, Comparison of the Farup and Schuma Methods for, 111

———, Nitroprusside Reaction of, 154

Urine, Proteolysed Substances, and Blood, α- and β-Amino Acids a Derivatives in, the Estimation of, Micro-Chemical Method for

———, Sugar (Dextrose) in, Estimation of, 65

———, Urea in, Estimation of, by Sodium Hypobromite, 269

———, Uric Acid in, Estimation of, 65

———, ———, the Estimation of, Colorimetric Method for, 154

Urine, Uric Acid in, Titration of, after Silver Precipitation, 329
Urobilin, Detection of, 463

Valve, New High-Pressure Reducing. Bomb with Stirring Gear for the Measurement of Reaction-Velocity in a Heterogeneous System at High Pressures, 590
alves, Gas Engine, Rapid Method of Testing Suction Gas Fuels for Liability to Clog, 3
anadium and Chromium in Steels, Determination of, 81
——, Chromium, and Iron, in Presence of One Another, the Volumetric Estimation of, a New Method for, 99
—— in Steel, Estimation of, by the Hydrogen Peroxide Colour Method, 589
—— Steel and Ferrovanadium, Phosphorus in, Determination of, 587
——, Steels Containing, Determination of Phosphorus in, 478
Extracts, Authentic, Chemical Composition of, together with Analytical hods and Limits of Constants, 206
es, Spirit, Analysis of. Part I. Investigation of the Solvent, 164
le and Animal Fats, Differentiation of, 458
— and Fish Oil Mixtures, Properties of. Studies on Fish Oils, 70
— Oils in Margarine, Detection of, 21
les, Water in, Estimation of, by Hoffmann's Apparatus, 177
y of Detonation, and of Brisance, Estimation of, 464
th, Saccharin in, Detection of, 22
— Wines, Composition of, 267
Table for Correcting the Specific Gravities of Milk, Extension of, 460
, Cider, Adulteration of, with Distilled Vinegar, Detection of, 565
neter, Precision, for Measurement of Relative Viscosity, and the Relative osities of Water at 0°, 18°, 25°, and 50° C., 399
neters, Redwood's and Engler's, Comparison of, 398
netry. Part II. Methods and Apparatus Used in Petroleum Testing, 397
y of Butter Fat and its Substitutes, 458
– of Lubricating Oils, 36
– of Rubber Solutions, Estimation of, 272
ol Seeds, Method for Detecting the, 370
e Fraction in Milk, Attempt to Estimate, 269
Blue, and Soda Ash, Mixtures of, Estimation of Sodium Carbonate and er in, by Titration with Potassium Hydroxide, 340
Constituents of Coal. Part III., 511
————. Part IV. Relative Inflammabilities of Coal Dusts, 512
—— of Roasted Coffee, 417
Fatty Acids, Detection of, 272
Products of Destructive Distillation, Water in, Estimation of, 348
Solvents, Improved Apparatus for Extracting Finely Divided Solids with,

ic Analyses, Mercuric Oxide as a Standard for, 439
— Determination, Direct, of Tin, 82
— ————, of Gold, 390
— Estimation of Chromium in Presence of Iron, 387
c Estimation of Hypophosphorous Acid and Hypophosphites, 342
— ———— of Manganese by the Volhard-Wolff Method. Chemiker kommission des Vereins deutscher Eisenhüttenleute, 391
— ———— of Phosphorus in Steel by Macagno's Method, 526

Volumetric Estimations, Use of the Sense of Smell in, 594
——— Method, New Physico-Chemical, 294

Washing Powders Containing Perborates, and Perborates, Active Oxygen in, Estimation of, 441
Water, Added, Detection of, in Milk, by Means of the Refractometer Value of the Serum, 500
———, Alcohols, and Potassium Fluoride, Systems Containing, Equilibria in. Method for Estimating Water in Alcohol, 425
——— Analysis, Bacteriological Methods of, with Special Reference to the Use of th Berkefeld Filter, 574
———, Colorimetric Methods for, by the Use of Autenreith-Koenigsber Colorimeter, 179
——— Potassium Palmitate in, Use of, 127
———, Baryta, Action of, on Certain Kinds of Glass, 118
——— *B. coli. communis* in, Estimation of, 574
———, Conductivity, Preparation of, 346
———, Determination of Hardness of, and Behaviour of Calcium and Magn Salts with Soap Solutions, 436
———, Distilled, Copper in, 474
———, Formed on Burning Substances Containing Nitrogen and Sulphur Calorimetric Bomb, Estimation of, 347
——— in Bleached Shellac, Estimation of, 280
——— in Cheese, Estimation of, 146, 322
——— in Foods, etc., Direct Estimation of, by Distillation, 207
——— in Petroleum and its Products, Estimation of, 165
——— in Silicate Minerals and Rocks, Estimation of, 47
——— in Vegetables, Estimation of, by Hoffmann's Apparatus, 177
——— in Volatile Products of Destructive Distillation, Estimation of, 348
———, Iron in, Estimation of, 174
———, "Raw" River, and Crude Sewage, Search for Pathogenic Microbes in
———, Small Quantities of Manganese in, Colorimetric Estimation of, 225, 4
———, Water-Soluble Phosphates in Ammonium Superphosphates, Estima 392
———, the Bacteriological Examination of, New Rapid Method for, and its cation for the Testing of Springs and Filter-Beds, 30
———, the Determination of Dissolved Oxygen in, Apparatus for, 396
———, ———————, Practical Field Method fo
———, the Estimation of Dissolved Oxygen in, Use of Sodium Potassium in, 442
———, the Permeability of Soils to, Determination of, 49
———, the Relative Viscosities of, at 0°, 18°, 25°, and 50° C., and Precision meter for Measurement of Relative Viscosity, 399
Waters, etc., Mineral, Iodine and Bromine in, the Estimation of, Hydrolyti a Source of Error in, 532
——— Lithium in, Estimation of, 531
——— Natural, Acidity and Alkalinity of, 40
——— Small Quantities of Hydrogen Sulphide in, Estim 532
———, ———, Various, Action of, on Lead, 530
Wax, Candelilla, 514
Wax, Paraffin, and Ceresin, in their Mixtures, Estimation of, 37
Waxes and Resins of Kapok and Akon Fibres, 580
Whale Oil, Hardened, Colour Reactions of, 433

Volumetric Estimations, Use of the Sense of Smell in, 594
———— Method, New Physico-Chemical, 294

Washing Powders Containing Perborates, and Perborates, Active Oxygen in, Estimation of, 441
Water, Added, Detection of, in Milk, by Means of the Refractometer Value of the Serum, 500
———, Alcohols, and Potassium Fluoride, Systems Containing, Equilibria in. Method for Estimating Water in Alcohol, 425
——— Analysis, Bacteriological Methods of, with Special Reference to the Use of th Berkefeld Filter, 574
——— ————, Colorimetric Methods for, by the Use of Autenreith-Koenigsb Colorimeter, 179
——— ————, Potassium Palmitate in, Use of, 127
———, Baryta, Action of, on Certain Kinds of Glass, 118
——— *B. coli. communis* in, Estimation of, 574
— —, Conductivity, Preparation of, 346
———, Determination of Hardness of, and Behaviour of Calcium and Mag Salts with Soap Solutions, 436
———, Distilled, Copper in, 474
———, Formed on Burning Substances Containing Nitrogen and Sulphu Calorimetric Bomb, Estimation of, 347
——— in Bleached Shellac, Estimation of, 280
——— in Cheese, Estimation of, 146, 322
——— in Foods, etc., Direct Estimation of, by Distillation, 207
——— in Petroleum and its Products, Estimation of, 165
——— in Silicate Minerals and Rocks, Estimation of, 47
——— in Vegetables, Estimation of, by Hoffmann's Apparatus, 177
——— in Volatile Products of Destructive Distillation, Estimation of, 348
———, Iron in, Estimation of, 174
———, "Raw" River, and Crude Sewage, Search for Pathogenic Microbes in
———, Small Quantities of Manganese in, Colorimetric Estimation of, 225, 4
———, Water-Soluble Phosphates in Ammonium Superphosphates, Estima 392
———, the Bacteriological Examination of, New Rapid Method for, and its A cation for the Testing of Springs and Filter-Beds, 30
———, the Determination of Dissolved Oxygen in, Apparatus for, 396
———, ————————————, Practical Field Method fo
———, the Estimation of Dissolved Oxygen in, Use of Sodium Potassium in, 442
———, the Permeability of Soils to, Determination of, 49
———, the Relative Viscosities of, at 0°, 18°, 25°, and 50° C., and Precision meter for Measurement of Relative Viscosity, 399
Waters, etc., Mineral, Iodine and Bromine in, the Estimation of, Hydrolyti a Source of Error in, 532
————, ————, Lithium in, Estimation of, 531
————, Natural, Acidity and Alkalinity of, 40
————, ————, Small Quantities of Hydrogen Sulphide in, Estim 532
————, Various, Action of, on Lead, 530
Wax, Candelilla, 514
Wax, Paraffin, and Ceresin, in their Mixtures, Estimation of, 37
Waxes and Resins of Kapok and Akon Fibres, 580
Whale Oil, Hardened, Colour Reactions of, 433

Bran and Cottonseed Meal, Phytic Acid in, 419
Metal Alloys, Copper in, Determination of, 222
Phosphorus in Match Compositions, Detection of, 289
———— in Phosphorus Sesquisulphide, Detection and Estimation of, 80
Chlorides in, Estimation of, 502
Colour Reaction of, 24
, Higher Alcohols in, Estimation of, 23
——, Malic, Tartaric, and Succinic Acids in, Estimation of, 502
——, Melnik (Bohemian), 326
——, Port, Composition of, 368
——, Quercitin in, Estimation of, 267
—— Statistics for the Years 1911-1912, 566
——, Succinic, Tartaric, and Malic Acids in, Estimation of, 502
——, Sulphates, Tartaric Acid, Calcium, and Magnesium in, Physico-Chemical Estimation of, 421
——, Tartaric, Succinic, and Malic Acids in, Estimation of, 502
Wines and Fermented Liquors, the Dry Extract of, Accurate Estimation of, 566
——— and Grapes, Estimation of Pentoses and Methylpentoses in; Investigations on Lead Precipitates from Wines, 25
———, Citric Acid in, Detection of, 55
———, Glycerol in, Estimation of, 368
———, Lead Precipitates from, Investigations on; Estimation of Pentoses and Methylpentoses in Grapes and Wines, 25
———, Musts, etc., Tartaric, Citric, Lactic, and Malic Acids in, Estimation of, 504
———, New Reaction for the Detection of Aniline Dyestuffs (Magenta) in Foods, and Especially in, 322
———, So-called Non-Alcoholic, of Commerce, 565
———, Sulphate in, Estimation of, 369
———, Sweet Raisin, Glycerol in, Estimation of, 24
———, the Bromine Absorption of, Estimation of, 266
———, Vermouth, Composition of, 267
Wires, Insulated, Rubber Wares, Particularly, Estimation of Caoutchouc in, 111
Wood, Mechanical, in Paper, the Detection of, Simple Reagent for, 434
"Wood Oils" from China and Japan, 580
Wood Turpentines, their Analysis, Refining, and Composition, 39
Wool Fat, Distilled, 106
—————, Oleins of, Detection of Mineral Oils in, 35
—————, Stearin of, 37
——— in Cotton Goods, Small Quantities of, Estimation of, 510

Xanthine Bases in Cocoa, Tea, and Coffee, and their Preparations, Detection and Estimation of, 26, 60
Ximenia Americana, the Seeds of, Oil from, 147

Yoghurt and Yoghurt Preparations, 31
Young's Method for the Preparation of Absolute Alcohol, Application of, 424

Zinc and Copper, Electro-Analytical Estimation of, Application of Tantalum trodes to, 481
—— as Zinc Ammonium Phosphate and Zinc Phosphate, Estimation of, 443
——, Copper, and Nickel, Solutions of, Action of an Excess of a Soluble Fer on, and a Volumetric Method for the Estimation of Nickel, 524
——, Gravimetric Estimation of, 47

Zinc, Group, Qualitative Analysis of the, 349
—— in Ores, Determination of, 298
—— in Treated Ties (Railway Sleepers), Estimation of, 589
——, Lead, and Copper, in Supply-Water, Detection and Colorimetric Esti of, 123
——, ——, and Nickel, Estimation of, by Precipitation as Oxalate and Ti with Permanganate, 122
—— Ores, Fluorine in, Estimation of, 475
——, Rapid Method of Estimating, 127
—— Salts, Precipitation of Purine Bases from Meat Extract and Urine by Means of, 572
————, Precipitation of Uric Acid and Purin Bases by, 424
——, Separation of Cadmium from, 383

JANUARY, 1913. Vol. XXXVIII., No. 442.

THE ANALYST.

INTERNATIONAL ATOMIC WEIGHTS, 1913.

		O=16.
Aluminium	Al	27·1
Antimony	Sb	120·2
Argon	A	39·88
Arsenic	As	74·96
Barium	Ba	137·37
Bismuth	Bi	208·0
Boron	B	11·0
Bromine	Br	79·92
Cadmium	Cd	112·40
Cæsium	Cs	132·81
Calcium	Ca	40·07
Carbon	C	12·00
Cerium	Ce	140·25
Chlorine	Cl	35·46
Chromium	Cr	52·0
Cobalt	Co	58·97
Columbium	Cb	93·5
Copper	Cu	63·57
Dysprosium	Dy	162·5
Erbium	Er	167·7
Europium	Eu	152·0
Fluorine	F	19·0
Gadolinium	Gd	157·3
Gallium	Ga	69·9
Germanium	Ge	72·5
Glucinum	Gl	9·1
Gold	Au	197·2
Helium	He	3·99
Holmium	Ho	163·5
Hydrogen	H	1·008
Indium	In	114·8
Iodine	I	126·92
Iridium	Ir	193·1
Iron	Fe	55·84
Krypton	Kr	82·92
Lanthanum	La	139·0
Lea	Pb	207·10
Lithium	Li	6·94
Lutecium	Lu	174·0
Magnesium	Mg	24·32
Manganese	Mn	54·93
Mercury	Hg	200·6

		O=16.
Molybdenum	Mo	96·0
Neodymium	Nd	144·3
Neon	Ne	20·2
Nickel	Ni	58·68
Niton (radium emanation)	Nt	222·4
Nitrogen	N	14·01
Osmium	Os	190·9
Oxygen	O	16·00
Palladium	Pd	106·7
Phosphorus	P	31·04
Platinum	Pt	195·2
Potassium	K	39·10
Praseodymium	Pr	140·6
Radium	Ra	226·4
Rhodium	Rh	102·9
Rubidium	Rb	85·45
Ruthenium	Ru	101·7
Samarium	Sa	150·4
Scandium	Sc	44·1
Selenium	Se	79·2
Silicon	Si	28·3
Silver	Ag	107·88
Sodium	Na	23·00
Strontium	Sr	87·63
Sulphur	S	32·07
Tantalum	Ta	181·5
Tellurium	Te	127·5
Terbium	Tb	159·2
Thallium	Tl	204·0
Thorium	Th	232·4
Thulium	Tm	168·5
Tin	Sn	119·0
Titanium	Ti	48·1
Tungsten	W	184·0
Uranium	U	238
Vanadium	V	the
Xenon	Xe	nt as
Ytterbium (Neoytterbium)	Yb	[illegible]
Yttrium	Yt	[illegible]
Zinc	Zn	e first
Zirconium	Z	extract

OBITUARY.

We regret to record the death on December 11, 1912, of Mr. H. de Mosenthal, technical secretary to Messrs. Nobel Dynamite Trust Co., Ltd.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

The monthly ordinary meeting of the Society was held on Wednesday evening, December 4, in the Chemical Society's Rooms, Burlington House. The President, Mr. L. Archbutt, F.I.C., occupied the chair.

The minutes of the previous ordinary meeting were read and confirmed.

Certificates of proposal for election to membership in favour of Messrs. S. Elliott, A.I.C.; T. R. Greenough, B.A., A.I.C.; H. Lowe, M.Sc.; J. P. Ogilvie; and J. A. L. Sutcliffe, A.I.C., were read for the second time; and certificates in favour of Messrs. John Augustus Goodson, F.I.C., Assistant Chemist to the Sudan Government in the Wellcome Research Laboratory, Gordon College, Khartoum; John C. White, A.I.C., Tudor Lodge, St. James's Road, Upper Tooting, Assistant Chemist to Messrs. Price's Patent Candle Co., Ltd.; and Frederick William Skevington, 87 and 89, Aldgate, E., assistant to Messrs. G. H. Ogston and Moore.

The following papers were read: "The Detection of Adulteration in Linseed Oil," by G. D. Elsdon, B.Sc., and Herbert Hawley, M.Sc.; "The Estimation of Citric Acid in the Presence of Certain Other Acids," by Lionel Gowing-Scopes; "The Value of the Guaiacum Test for Bloodstains," by Herbert S. Shrewsbury, F.I.C.; "The Determination of Nitrates and Nitrites in Sewage Effluents," by Arnold Higginson; and "Alcoholysis and the Composition of Cocoanut Oil," by G. D. Elsdon, B.Sc.

The President announced the Council's nominations of officers and Council for 1913 as follows:

President.—L. Archbutt, F.I.C.

Past-Presidents (limited by the Society's Articles of Association to eight in number).—Edward J. Bevan, F.I.C.; Bernard Dyer, D.Sc., F.I.C.; Thomas Fairley, F.I.C.; W. W. Fisher, M.A., F.I.C.; Otto Hehner, F.I.C.; R. R. Tatlock, F.I.C.; E. W. Voelcker, A.R.S.M., F.I.C.; J. Augustus Voelcker, M.A., B.Sc., Ph.D., F.I.C.

Vice-Presidents.—G. Embrey, F.I.C.; J. T. Hewitt, M.A., D.Sc., Ph.D., F.R.S. W. H. Willcox, M.D., B.Sc., M.R.C.P., F.I.C.

Hon. Treasurer.—Edward Hinks, B.Sc., F.I.C.

Hon. Secretaries.—A. Chaston Chapman, F.I.C.; P. A. Ellis Richards, F.I.C

ier Members of Council.—R. M. Clark, B.Sc., F.I.C.; J. Connah, B.Sc., F.I.C.; s, F.I.C.; G. T. Holloway, A.R.C.Sc., F.I.C.; G. D. Lander, D.Sc., F.I.C.; ra, F.I.C.; G. W. Monier-Williams, M.A., Ph.D., F.I.C.; C. Revis; H. B.Sc., F.I.C.; F. Wallis Stoddart, F.I.C.; A. R. Tankard, F.I.C.; John

THE DETECTION OF ADULTERATION IN LINSEED OIL.

By G. D. ELSDON, B.Sc., A.I.C., and HERBERT HAWLEY, M.Sc., A.I.C.

(*Read at the Meeting, December* 4, 1912.)

One of us and another, Liverseege and Elsdon (*J. Soc. Chem. Ind.*, 1912, **31**, 207) recently described a modified form of the Livache test, which was capable of giving quantitative figures for the oxygen absorbed by oils on exposure to the atmosphere. The close relationship existing between the iodine value and the figure thus obtained was pointed out, but we have since found that this is of more importance than at first appeared to be the case. We thought that an indication of the purity of a linseed oil might be obtained by finding the relationship between the oxygen absorbed and the iodine value; but, although this relationship is close, we found that the differences between linseed oil and other oils in this respect were not sufficient to detect small adulterations. The method was accordingly abandoned.

The finding of a figure which would have a fixed relationship to the iodine value seemed to promise good results, and it appeared to us that further figures might be obtained by an analysis of the oxidised residue obtained in the above process. For this purpose extraction with ether on quantitative lines naturally suggested itself, the oxidation products of oils being insoluble in this solvent. The difficulty, however, of transferring the hard paste to an extraction thimble, and the readiness with which the finely divided litharge passed through the paper into the flask, complicated this method to such an extent that it was finally discarded in favour of drying on Adams' coils.

2·5 grms. of the oil are accurately weighed out into a small, wide-mouthed flask, and transferred by means of ether (using a small funnel) to a 25 c.c. graduated flask—those which are used for roughly taking specific gravities serve admirably. The solution is then diluted to the mark with more ether, and the whole thoroughly mixed. Five c.c. of this solution are then transferred by means of a carefully-dried pipette to an Adams' coil (the fat-free strips made by Schleicher and Schull for the estimation of fat in milk), care being taken that the solution is uniformly distributed over the whole paper. After drying, by whatever method (see below), the strip is rolled up and placed in a Soxhlet fat-extraction apparatus and extracted with ether (sp. gr. 0·720) for three hours. At the end of this time the ether is evaporated, a little absolute alcohol added, and the flask dried in the steam oven for two hours; it is then cooled and weighed.

Drying in the Air at Laboratory Temperature.—The first experiments were made using the above process, and allowing the oil-saturated strips to dry in the air of the laboratory for varying lengths of time, the extraction then being carried out as described above. Some results obtained in this way are given in Table I.

It will be seen from this table that the drying proceeds rapidly during the first week, then more slowly, and is practically constant after a fortnight, the extract then showing a slight tendency to increase.

TABLE I.

Oil.	Iodine Value. (Wijs.)	Extract per Cent.			
		3 Days.	1 Week.	2 Weeks.	3 Weeks.
Linseed oil A	194	46·4	26·0	20·2	20·6
„ B	179	76·4	33·4	29·8	—
Colza oil	99	—	100·6	100·8	101·2
Linseed oil A with 20 per cent. colza	176	—	36·8	29·6	30·0

The reason for this increase in the extract is not particularly obvious, but it would seem to be intimately connected with the loss in weight which occurs in the above-mentioned Livache process after the maximum gain has been reached. Possibly a portion of the oxygen is loosely combined, and after the maximum quantity has been absorbed some slight decomposition takes place, substances soluble in ether being formed, and oxygen or some other gas being evolved.

Drying in the Water Oven.—Although the figures obtained by drying in the air are fairly concordant, the time required is obviously too long for a laboratory test. Our final modification shortens the process considerably, at the same time giving concordant results. The process is as follows:

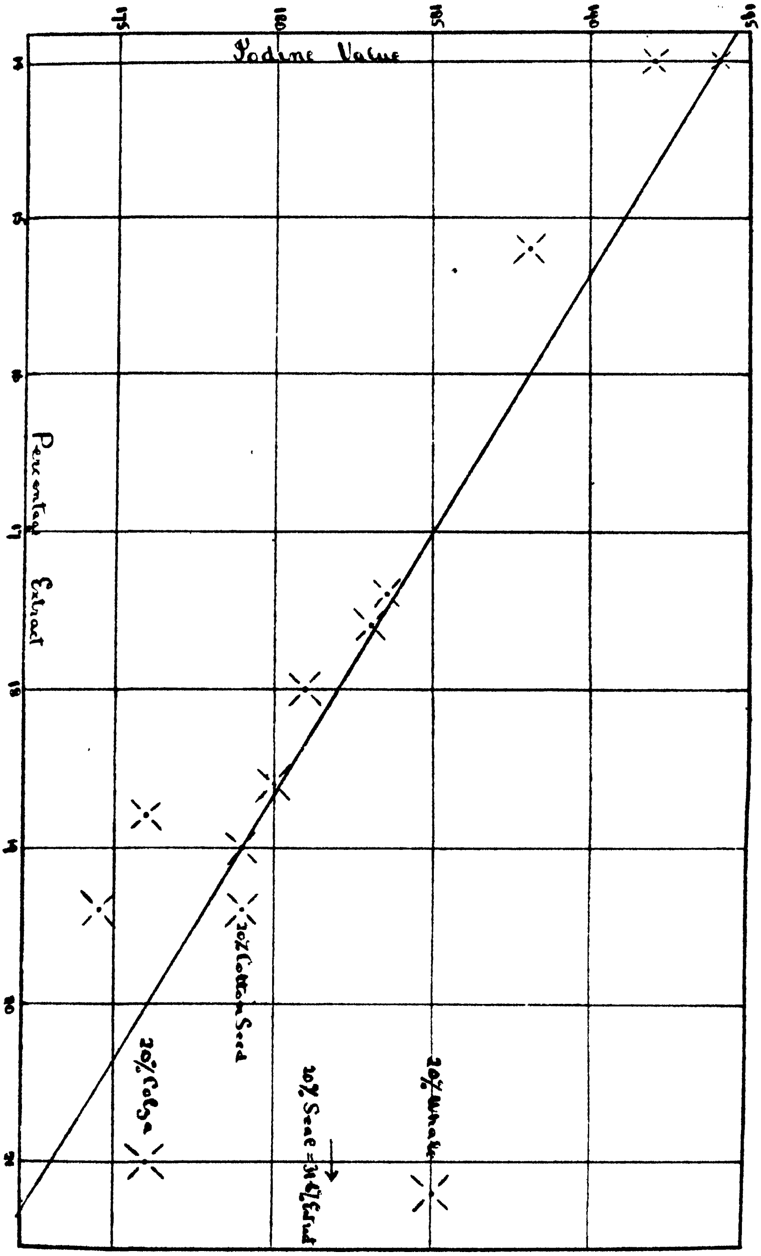
The oil is distributed on the coils as described above, and these are allowed to dry in the air overnight, the drying being then continued in the steam oven for varying lengths of time. The strips are folded three times, and so arranged on their edges in the oven that the whole of the surface is exposed. The extraction of the dried coils is then carried out in the usual manner. In carrying out this method it is necessary to have a fairly large oven (12 inches cube), and to place the strips at the bottom and well away from the door, the shelves being removed to give plenty of air-space above the coils. Some results obtained by this method are shown in Table II.

TABLE II.

Oil.	Iodine Value. (Wijs.)	Extract per Cent.				
		2 Hours.	4 Hours.	8 Hours.	17 Hours.	41 Hours.
Linseed oil A	194	14·0	13·6	13·2	10·0	9·4
„ B	179	19·0	17·6	16·2	13·2	10·4
Colza oil	99	100·6	100·2	—	74·0	40·0
A + 20 per cent. colza ...	176	21·0	19·0	18·8	14·8	11·0
A + 20 per cent. whale...	185	21·2	—	—	—	—

From a consideration of these and similar results it was decided that a period of two hours in the boiling-water oven was most convenient, with subsequent extraction for three hours as described above.

It will be observed that the addition of 20 per cent. of either whale or colza oil to the linseed oil A makes a considerable difference in the extract; but, owing to



the variation in the extracts obtained from different linseed oils, this cannot be made use of as a quantitative test (*cf.* Table III.).

In Table III. are given the results of the examination of several pure oils and known mixtures, together with the iodine values (Wijs), modified Livache figures, and Zeiss butyro-refractometer readings at 25° C.

It will be seen from Table III. that a close relationship exists between the iodine value and the extract obtained by the method described; this relationship, however, becomes even more marked when the values are plotted as in the diagram.

A consideration of the results shows that the point produced by plotting the iodine value of a pure linseed oil against the extract should fall to the left of the dotted line. Now, in the case of many mixtures of the oils the iodine values and the extracts might be quite passable when taken alone, yet, when plotted in this way, the point would fall to the right of the dotted line, and the oil would be shown to be suspicious and to require further examination. Thus the linseed mixtures containing 20 per cent. respectively of cottonseed, colza, seal, and whale lie well outside the line, so that, although these might be passed as genuine linseed on either the extract or the iodine value alone, yet, when they are considered together, the oils are seen to be obviously adulterated.

TABLE III.

Oil.	Iodine Value. (Wijs.)	Livache (Modified).	Extract per Cent. 2 Hours.	Zeiss. 25° C.
Linseed	192	—	14·0	82·2
,, ...	174	13·3	19·2	79·0
,, ...	194	17·4	14·0	83·0
,, ...	179	14·6	19·0	79·6
,, ...	183	15·5	17·4	80·6
,, ...	188	16·3	15·2	82·4
,, ...	183	15·3	17·6	80·3
,, ...	180	15·2	18·6	80·8
,, ...	176	13·8	18·8	78·9
,, ...	181	15·2	18·0	80·8
Colza	99	—	100·6	66·0
Linseed + 20 per cent. colza ...	176	13·5	21·0	79·7
,, + 20 per cent. whale ...	185	14·9	21·2	—
,, + 20 per cent. cottonseed ...	179	15·1	19·4	79·4
,, + 20 per cent. seal... ...	181	16·3	31·6	—

The following equation has been calculated from the curve for the maximum extract of a linseed oil. If I is the iodine value, then the maximum permissible extract for that oil is given by—

$$\text{Extract} = 81{\cdot}9 - 0{\cdot}35\ \text{I}.$$

It is suggested that the routine examination of linseed oils be restricted to the determination of the iodine value, and the extract as described above, by which means decisive information as to the purity or otherwise of any sample may be obtained.

CITY ANALYST'S LABORATORY,
BIRMINGHAM.

DISCUSSION.

Mr. L. M. **NASH** thought that the authors might perhaps have chosen better examples of adulterants than they had mentioned. Soya oil, sunflower oil, perilla oil, and wood oil, were more likely to be used, and would be less easy to detect by specific tests. An admixture of 20 per cent. of colza oil would be shown at once by its effect on the viscosity.

Mr. R. **BODMER** said that, in examining samples of linseed oil by the bromine addition method, he had found that erratic results were sometimes obtained, owing apparently to differences in the mode of working. The method as laid down by Messrs. Hehner and Mitchell consisted in dissolving the oil in cooled ether, adding the bromine, and then, after allowing the precipitate to subside, washing four times with 10 c.c. of cooled ether; while Dr. Lewkowitsch, in an early edition of his book, recommended washing with alcohol and acetic acid. The results obtained by the two modes of procedure were in his (the speaker's) hands quite different, and yet the figures laid down by the respective authors for linseed oil were the same. Evidently, in order to insure concordant results, the mode of procedure must be absolutely uniform.

Dr. **RIDEAL** remarked that the results would probably have been more interesting had they embraced smaller admixtures—say 10 per cent.—of the foreign oils. The positions of the crosses on the authors' diagram were somewhat difficult to understand. In the case of pure linseed oil one would expect the ratios between the iodine value and the percentage of extract to fall into a straight line, or at any rate that the number of crosses inside and outside the line would be the same.

Mr. E. R. **BOLTON** remarked that, in the case of an oil containing a considerable proportion of oxidisable substances, some of the extractable matter would probably be retained by the oxidised oil, just as in the Adams' process for determining fat in milk some of the fat was liable to be held back by the casein.

Mr. **CRIBB** said that possibly the difficulty to which Mr. Bolton had alluded might be avoided by the use of thinner paper. The very thin "Japanese" paper used for press-copying answered well, allowing the oil to be spread in a much thinner film.

The **PRESIDENT** said that he had found Messrs. Hehner and Mitchell's process to be a very valuable one, though he quite agreed with Mr. Bodmer that it was necessary to work under uniform conditions. The question as to what conditions were the best for obtaining uniform results appeared still to need investigation, and he thought that this was a matter which might usefully be taken up under the Society's Analytical Investigation Scheme.

Mr. N. **EVERS**, in the absence of the authors, said that most of their determinations had been made in duplicate, the duplicate results agreeing fairly well. The film of oxidised oil undoubtedly prevented some of the non-drying oil from being extracted—in fact, if that were not the case, the results obtained by extraction would be quantitative.

ALCOHOLYSIS AND THE COMPOSITION OF COCOANUT OIL.

BY G. D. ELSDON, B.Sc., A.I.C.

INVESTIGATION CARRIED OUT UNDER THE SOCIETY'S ANALYTICAL INVESTIGATION SCHEME.

(*Read at the Meeting, December* 4, 1912.)

THE full details of the process of alcoholysis were first published by A. Haller in 1906 (*Compt. rend.*, 1906, **143**, 657 ; ANALYST, 1907, **32**, 52), but the same idea had been previously used by Fox and Wanklyn, who, some years before (*ibid.*, 1884, **9**, 73), described a method for the analysis of butter which depended upon the formation of ethyl butyrate on heating with alcoholic potash. The method of Haller has been used for determining the composition of various oils and fats, and the following papers have been published on these lines : "Alcoholysis of Cocoanut Oil," Haller and Youssoufian (ANALYST, 1907, **32**, 53); "Alcoholysis of Castor Oil," Haller (*J. Soc. Chem. Ind.*, 1907, **26**, 328); "Alcoholysis of Linseed Oil," Haller (*ibid.*, 1908, **27**, 234); "Alcoholysis of Japan Wax," Tassily (*ibid.*, 1911, **30**, 907); "Alcoholysis of Cottonseed Oil," Meyer (*Chem. Zeit.*, 1907, **31**, 793); "The Determination of the Composition of Complex Lipoids by Alcoholysis," Fourneau and Piettre (ANALYST, 1912, **37**, 463). In none of these cases, however, has the method been made quantitative, and it was therefore suggested to the author, through the medium of the Analytical Investigation Scheme, that an examination of the process should be made with special reference to its quantitative applications, with a view, if possible, of finding a method for distinguishing between cocoanut oil and palm-kernel oil. The results obtained from the alcoholysis of cocoanut oil are given below; palm-kernel oil is at present being examined, and the author hopes to publish the results in due course.

In the case of cocoanut oil it is proposed to describe the process used and the results obtained under two headings: (1) Alcoholysis, and (2) the composition of cocoanut oil.

1. ALCOHOLYSIS.—For these experiments methyl alcohol has been used exclusively, on account of the fact that the methyl esters of the fatty acids have lower boiling-points than the corresponding ethyl esters. The process, which consists in boiling the oil with excess of methyl alcohol, will now be described in detail.

It has been shown by Patterson and Dickinson (*J. Chem. Soc.*, 1901, **79**, 280) that when any ester is heated with excess of another alcohol the two alcohols change places, and the acid of the ester combines solely with the alcohol in excess. Thus, on heating ethyl tartrate with excess of methyl alcohol, the whole of the ethyl tartrate is transformed into methyl tartrate. From this it was surmised that "pure" commercial methyl alcohol might be used in place of the absolute alcohol used by Haller, as any higher alcohols present as impurities would not be transformed into esters in he presence of such a large excess of methyl alcohol, and the effect of such substances as acetone would merely be to assist alcoholysis. In all the experiments described in this paper this commercial alcohol has been used with satisfactory results.

The alcohol was prepared for use as follows: "Pure" commercial methyl alcohol was dried over lime for several days with the help of a shaking apparatus; it was then roughly filtered and distilled from the water-bath, the fraction 65° - 72° C. being collected. A current of dry hydrochloric acid gas was then passed through the distillate until it had increased by about 2 per cent. of its weight.

The Process of Alcoholysis.—The process used is based on that of Haller (*loc. cit.*), and is as follows: Five hundred grms. of dry coconut oil were placed together with 1200 c.c. of methyl alcohol (obtained as described above) in a conical flask, and the whole heated on the water-bath until it had become clear, this process taking ten to fifteen hours; it was then heated for about ten hours more to make sure that the reaction was complete.

The mixture, on cooling, deposited a large quantity of esters, and these were removed as completely as possible by means of a separating funnel; the alcoholic layer was then poured into at least an equal volume of water, and the whole extracted four times with ether, the ethereal solution being added to the separated esters. The alcoholic layer was disregarded.

The ethereal solution of esters was then twice washed with a dilute solution of sodium carbonate, and then twice with water in order to remove hydrochloric acid, and alcohol; it was then dried over fused sodium sulphate. The ether was finally removed by heating in a water-bath until nothing further distilled.

The esters obtained from two such experiments (representing 1 kgrm. of cocoanut oil) were then fractionally distilled in quantities of about 250 grms. at a time, under a pressure of 14 mm., the corresponding distillates obtained in the different distillations being mixed together and finally weighed. The receiver was changed during the progress of the distillation at a temperature midway between the boiling-points of two consecutive esters. For convenience of reference a table of the melting and boiling points of the methyl esters of the chief fatty acids likely to occur in oil and fats is given below:

Name.	Melting-Pt. ° C.	Boiling-Pt. ° C.	
		760 mm.	15 mm.
Methyl n. butyrate	—	102	—
n. valerate	—	117	—
n. caproate	—	150	52·5
n. caprylate	- 40·5	194	83·0
n. caprate	- 18·0	224	114·0
n. laurate	+ 5·0	—	141·0
n. myristicate	+ 18·0	—	167·5
n. palmitate	28·0	—	196·0
n. stearate	38·0	—	214·5
oleate	—	—	212·5

The individual fractions thus obtained, whose weights will be roughly proportional to the quantities of the various methyl esters present in the oil, are then refractionated until a fraction is obtained of constant boiling-point. In this way the various constituents of the oil are isolated as their methyl esters, and the individual esters can then be identified by physical and chemical means.

The separation of methyl oleate from methyl stearate requires a special process, as the boiling-points of the two are so nearly alike. It is carried out, as described by Haller, by cooling the mixture in ice, and filtering the solid methyl stearate from the liquid methyl oleate. This part is one of the weak points of the process, as it is by no means easy to separate them in this way; and, indeed, complete separation is an impossibility.

The Results obtained.—From the experiments carried out it has been concluded that the process of alcoholysis is a suitable one for determining the qualitative composition of an oil, and that it also gives considerable information in regard to the quantities of the constituents present; the process, however, is much too lengthy for use as an ordinary laboratory test, and would seem to be chiefly of theoretical interest. The quantitative results have not been encouraging, but the process probably gives results within about 5 to 10 per cent. of the true value—that is to say, it should decide between 35 and 45 per cent. for the content of lauric acid in cocoanut oil.

2. THE COMPOSITION OF COCOANUT OIL.—The composition of cocoanut oil has been the subject of various papers, but the results given by the various workers, while having much in common, show greater divergences than would be expected from the variation in the composition of different samples.

It has been shown by Jensen (ANALYST, 1905, **30**, 397) that cocoanut oil contains no butyric acid and but little caproic, the volatile acids being mainly capric and caprylic. Paulmyer (*J. Soc. Chem. Ind.*, 1907, **26**, 881) found that the fatty acids of cocoanut oil consisted of capric, 20 per cent.; lauric, 40 per cent.; myristic, 25 per cent.; palmitic, 11 per cent.; and oleic, 5 per cent.; together with capric and caprylic acids; while Caldwell and Hartley (ANALYST, 1909, **34**, 274) found by a different method at least 60 per cent. of lauric acid. Haller and Youssoufian found, by the method of alcoholysis, caproic, caprylic, capric, lauric, myristic, palmitic, stearic, and oleic acids. They did not give any quantitative results, but they stated that lauric and myristic acids predominated, whilst palmitic, stearic, and oleic were present in relatively small quantities. It will be found that these results of Haller have been largely confirmed by the results obtained by the author, and which are given below.

The sample of cocoanut oil used in the experiments described in this paper had the following analytical constants, and was in all respects a fair average commercial sample. The figures obtained are in close agreement with those obtained by Haller on the sample used by him:

Saponification equivalent	25·84
Iodine value (Wijs)	8·71
Melting-point	24° C.
Reichert-Meissl value	7·71
Zeiss at 40° C.	34·90

One kgrm. of the oil was used for alcoholysis, and from this 976 grms. of esters were obtained. These were fractionally distilled under reduced pressure (14 mm.) as described above, and the following fractions were obtained:

Boiling-Pt.	Weight.	Corresponding to—
63° to 76° C.	16·5 grms.	Methyl caproate
76° „ 100° C.	85·0 „	„ caprylate
100° „ 128° C.	92·5	caprate
128° „ 153° C.	442·0	laurate
153° „ 182° C.	206·0	myristicate
182° „ 204° C.	66·5	palmitate
204° „ 216° C.	67·5	stearate and oleate

These various fractions were then refractionated, and each ester was obtained in the pure state, or as nearly pure as the quantity of the matter would allow. They were then examined by physical and chemical means, and each ester mentioned above identified with certainty. About 20 grms. of methyl oleate were obtained, but the subsequent purification was difficult. No indications were obtained of the presence of any other fatty acids but those mentioned.

From a consideration of these results, the composition of the fatty acids of cocoanut oil would appear to be somewhat as follows, but, as already stated, the figures are rough, and must only be taken as an indication of the relative amounts present:

Caproic acid	2 per cent.
Caprylic acid	9
Capric acid	10
Lauric acid	45
Myristic acid	20
Palmitic acid	7
Stearic acid	5
Oleic acid	2
	100 per cent.

In conclusion, it is with great pleasure that I acknowledge the valuable assistance which I have received from Mr. A. Chaston Chapman and Dr. J. Lewkowitsch, both in connection with the above work and with that on palm-kernel oil at present being carried on. Part of the cost of the investigations has been defrayed by a grant from the Society.

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* * * * *

THE ESTIMATION OF CITRIC ACID IN THE PRESENCE OF CERTAIN OTHER ACIDS.

BY L. GOWING-SCOPES.

INVESTIGATION CARRIED OUT UNDER THE SOCIETY'S ANALYTICAL INVESTIGATION SCHEME.

(*Read at the Meeting, December* 4, 1912.)

UNTIL quite recently the only method available for the estimation of citric acid consisted in precipitating the acid as tricalcium citrate. It has been shown, however, by O. von Spindler (*Chem. Zeit.*, 1903, **27**, 1263), that this method is quite unreliable. Denigès (*Revue Général du Lait*, vol. iii., p. 385) devised a method by which the citric acid is oxidised to acetone-dicarboxylic acid, and precipitated as an insoluble mercury compound. This process has been examined by M. Beau (*Annales de Chim. et de Phys.*, 1899, 18), L. Robin (*Ann. de Chim. anal.*, 1904, **9**, 453), and E. Desmoulièrè (*Bull. d. Sciences Pharmacol.*, 1910, **17**, 588). L. Robin states that Denigès' test has not proved satisfactory in his hands, and proposes a method based on the precipitation of citric acid as cadmium citrate. E. Desmoulièrè finds Denigè's test useless for quantitative purposes. M. Beau, however, appears to have obtained satisfactory results, and his modification appears in Barthel's "Milk and Dairy Products." In addition to these methods, M. Spica (*Chem. Zeit.*, 1910, **34**, 1141) proposed to precipitate the acid as calcium citrate, digest with strong sulphuric acid, and to measure the carbon monoxide evolved. D. S. Pratt (*U.S. Dept. of Agriculture Bureau Chem.*; through *Chem. Eng.*, 1912, **15**, 72) oxidises to acetone, distils into mercuric sulphate, boils with this reagent, and weighs as an addition product. The present investigation was undertaken to examine critically Beau's modification of Denigès' method.

The principle on which the process is based is that when citric acid is oxidised with potassium permanganate, acetone-dicarboxylic acid, carbon dioxide, and water are formed.

$$C_6H_8O_7 + O = C_5H_6O_5 + CO_2 + H_2O.$$

Acetone-dicarboxylic acid on treatment with excess of mercuric sulphate gives a white precipitate of mercury dicarboxysulphoacetone. The precipitate, which is insoluble in water, is dissolved in hydrochloric acid, excess of ammonia added, as well as a known quantity of $\frac{N}{10}$ potassium cyanide, and then titrated with $\frac{N}{10}$ silver nitrate, using potassium iodide as indicator. Beau precipitates the casein in milk with Denigès' mercuric sulphate reagent, filters, and estimates the citric acid in the filtrate in the following manner: 100 c.c. of the filtrate are oxidised with a 1 per cent. solution of potassium permanganate, the liquid is heated to boiling; the flame is then removed and permanganate solution added drop by drop, the flask being shaken after each addition. The liquid soon becomes turbid, after which a yellowish-white precipitate forms. The addition of the potassium permanganate is, however, continued until the precipitate sinks rapidly to the bottom and the supernatant liquid is perfectly clear. To reach this point about 5 to 10 c.c. are required. A slight excess is of no disadvantage, for otherwise the oxidation is apt to be incomplete. The precipitate has a coffee-brown colour on account of small quantities of manganese dioxide, but this can be removed by heating the liquid until it boils, removing the flame, and adding small amounts of hydrogen peroxide until the precipitate is quite colourless. Generally 5 to 10 drops of hydrogen peroxide are required for this. The precipitate is then filtered off, dissolved in hydrochloric acid, and titrated as above.

These directions were followed, with the exception that instead of titrating, the precipitate was filtered through a Gooch crucible, and dried to constant weight. The results varied considerably. The effect of adding standard amounts of reagents, boiling for a definite time, and filtering, etc., was tried, but the results continued to vary for some undetermined reason.

Other oxidising agents were experimented with. Nitric acid either did not oxidise the citric acid to acetone-dicarboxylic acid or kept the precipitate in solution. Potassium bichromate gave a precipitate of an ochreous colour, but it was readily oxidised further and its use was abandoned. O. von Spindler (*Chem. Zeit.*, 1904, **28**, 15) has used potassium bichromate successfully as a qualitative test, and states that the precipitate contains chromium as well as mercury. Bromine proved useless, as also did ferric chloride. Ammonium persulphate and hydrogen peroxide gave pure white precipitates, which separated well from boiling solutions; but when employed quantitatively conflicting results were obtained, the ratio $\frac{\text{weight of precipitate}}{\text{weight of citric acid taken}}$ approximating to 3·5.

It was found that hydrogen peroxide oxidised the precipitate at 100° C., and even at lower temperatures the results were unsatisfactory. A number of experiments were made at temperatures varying from 16° to 53° C., the same amount of citric acid being used in each case. The precipitates were filtered, dried, and weighed at hourly intervals. The weight of the precipitate gradually increased to a maximum, then fell fairly rapidly, and when kept at a temperature of 53° C. in the course of a few days entirely disappeared. It appears that the precipitate is no sooner formed than it is oxidised again, and this happens before the whole of the citric acid is precipitated. These experiments explain the reason of the variable nature of Beau's results (*loc. cit.*); when the manganese dioxide is removed with hydrogen peroxide part of the precipitate is oxidised to acetone.

Hydrogen peroxide having proved a failure, the use of potassium permanganate was further investigated, and for the removal of manganese dioxide several substances were tried in place of hydrogen peroxide; some, such as oxalic acid, were too powerful reducing agents and decomposed part of the precipitate, whilst others were not strong enough to remove the manganese dioxide. After many trials fairly good results were obtained with a solution of "formalin." The citric acid was oxidised at 100° C. with potassium permanganate until the precipitate was slightly brown, formalin was added, and the mixture boiled until the precipitate appeared quite white. The following table gives the ratio $\frac{\text{weight of precipitate}}{\text{weight of citric acid taken}}$ for several sets of experiments made:

Citric Acid alone.			In the Presence of other Acids.	
First Set.	Second Set.	Third Set.	Acid.	Ratio.
3·55	3·83	3·57	Succinic acid	3·94
3·65	3·85	3·48	Tartaric acid	5·29
3·50	3·84		Lactic acid	3·93
	3·77		Citric acid alone ... (control experiment)	3·92

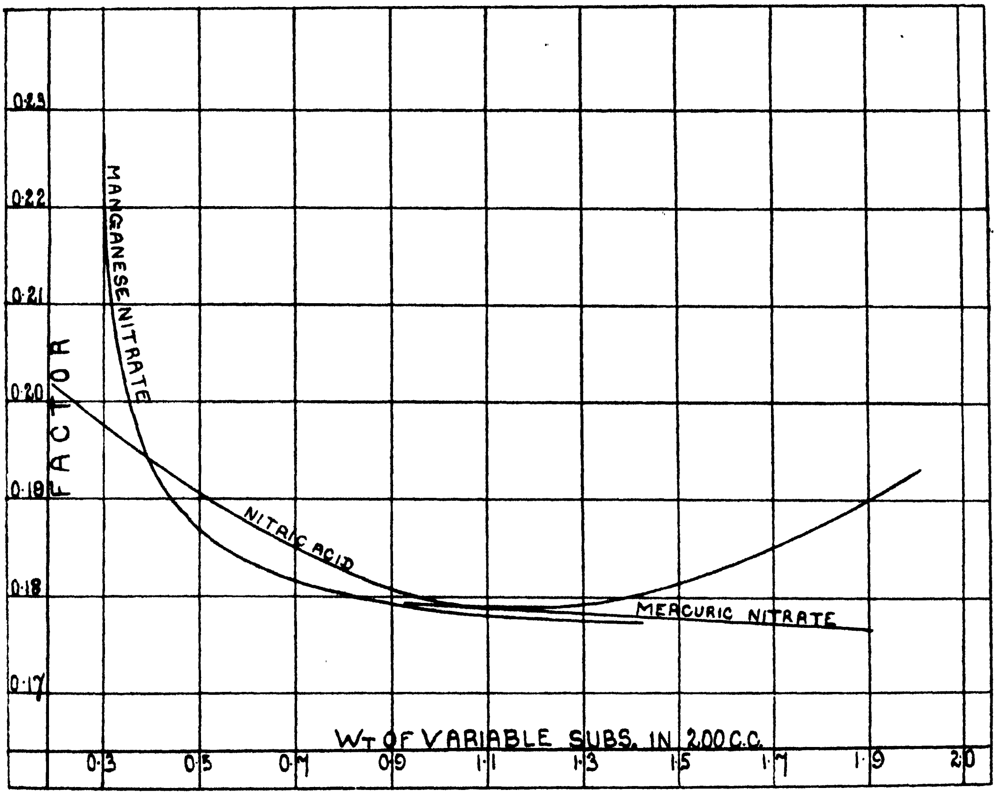
The other acids used were present to the extent of about three times the weight of citric acid. It will be seen that the ratio varies from about 3·5 to about 3·9, a variation sufficient to cause a fairly large error. It appears that the reason is due to the oxidising action of the precipitated manganese dioxide on the precipitate. Attempts to dissolve and reprecipitate, to prepare halogen derivatives, or to titrate the solution with iodine were unsuccessful. Better results were obtained by titrating the hydrochloric acid solution with potassium bromate, but these varied considerably. Incidentally it was found that methylene blue formed an excellent indicator for bromine, acting in the same manner as it does with iodine, as was first shown by Sinnat (ANALYST, 1910, **35**, 309).

As acetone-dicarboxylic acid is a strong reducing agent, it is possible that solutions of the precipitate might reduce ferric chloride, but this was not the case, or at any rate the oxidation was too slow to be of any use. Unsuccessful attempts were made to prepare derivatives, using for this purpose phenylhydrazine and its derivatives—semicarbazide, sodium bisulphite, etc.

It was thought that the small quantity of nitrous acid in Wiley's mercuric nitrate solution was affecting the results, and the solution was therefore treated with potassium permanganate till a precipitate was formed which was removed. The oxidised solution was then added to some citric acid, and the whole was heated to boiling ready to add permanganate to oxidise the citric acid and so obtain Denigès' precipitate. Instead of the liquid remaining clear when boiled a white precipitate rapidly formed. Further investigation proved that the cause was the manganese nitrate formed in the oxidised Wiley's solution, and much better results were obtained by using a mixture of manganese nitrate and mercuric nitrate in a nitric acid solution. This is the principle on which the method now proposed as an alternative to Denigès' method is based.

In order to ascertain what proportions of manganese nitrate, mercury nitrate, and nitric acid to use, experiments were made in which the proportion of two of the reagents was kept constant and that of the third varied. As it seemed probable that the nitric acid would have a solvent action on the precipitate, this was varied first. The citric acid taken was about 0·027 grm., the manganese nitrate

1·25 grms., and the mercuric nitrate 0·95 grm. The mixture was made up to 200 c.c. and boiled under a reflux condenser for three hours, filtered through a Gooch crucible, and the precipitate weighed. The variation in the factor for different amounts of nitric acid is shown in the curve. In the same way the manganese nitrate was varied while the citric acid and the nitric acid were kept constant. The amount of nitric acid taken corresponded to the maximum precipitate on the curve. In a like manner the mercuric nitrate was varied. Both results are shown by curves. When, however, the citric acid was varied, it was found that the larger the amount of citric acid taken the smaller the precipitate. This seemed to suggest that the mercury reagent should bear some relation to the amount of citric acid present. As the amount of citric acid present is generally unknown, the only way appeared to be to add neutral mercuric nitrate until the precipitate formed just dissolved, and, as the curves showed that about equal quantities of the various reagents should be present, amounts of manganese nitrate and nitric acid equivalent to the amount of mercuric nitrate were added, and the whole was diluted so that each constituent was present to the extent of 1 per cent. The mixture was then boiled for three hours, precipitated, filtered, dried, and weighed in the usual manner. By adopting this procedure, consistent results, as shown in the following table, were obtained:



Weight of Citric Acid taken.	Weight of Precipitate obtained.	Factor.
0·0028	0·0170	0·1647
0·0112	0·0684	0·1637
0·0196	0·1217	0·1612
0·0280	0·1700	0·1647
0·0055	0·0347	0·1585
0·0083	0·0503	0·1650
0·0153	0·0932	0·1642
0·0236	0·1412	0·1671

Experience gained by using this method showed that if large quantities of the reagent were used better results were obtained. Experiments were then made to see if this was the cause of the fall in the factor, as the citric acid was increased in the earlier experiments. Three grms. of each of the reagents, mercuric nitrate, manganese nitrate, and nitric acid, were used for each experiment. The citric acid was varied and the solution made up to 300 c.c. and boiled for three hours. Constant results were obtained, which were in excellent agreement with those obtained by the previous method. They are given in the following table :

Weight of Citric Acid taken.	Weight of Precipitate obtained.	Factor.
0·0027	0·0160	0·1688
0·0138	0·0840	0·1643
0·0202	0·1220	0·1656

In the next series 2 grms. of each reagent were taken, the citric acid was varied as before, and the solution was diluted to 200 c.c. The results obtained again agreed well with the foregoing, and this strength of solution was ultimately adopted.

A reagent containing mercuric nitrate, manganese nitrate, and nitric acid was prepared in the following manner: Fifty-one grms. of mercuric nitrate and 51 grms. of manganese nitrate were covered with about 68 c.c. strong nitric acid. About 100 c.c. of water were added, and when the salts were dissolved the volume was made up to 250 c.c. and filtered. Ten c.c. were taken for each estimation, corresponding with 2 grms. of each ingredient.

The estimation is carried out as follows: A quantity of the substance, containing not more than 0·04 grm. nor less than 0·001 grm. of citric acid, is exactly neutralised with $\frac{N}{10}$ alkali, using phenolphthaleïn as indicator. Ten c.c. of the reagent are added, and the whole diluted to 200 c.c. The mixture is boiled under a reflux condenser for three hours, filtered through a weighed Gooch crucible, and the precipitate well washed with cold water. Usually there is a deposit on the sides of the flask which may be removed by adding 1 or 2 c.c. 1 per cent. nitric acid, and rubbing with a rod. The precipitate is washed first by decantation and then in the crucible, and is dried in the water-oven for five hours, when the weight will be found to be nearly constant. Further drying usually only reduces the weight by less than 1 mgrm. The precipitate is white, with a slight cream tinge, if it has been prepared properly. If it is at all yellow, basic salts have formed, and the estimation will be high. The following table gives the results obtained by using pure citric acid, the exact amount being determined by titration:

Weight of Citric Acid taken.	Citric Acid found (using Factor 0·01667).	Difference.	Weight of Precipitate.	Factor.
0·0027	0·0027	0·0000	0·0162	0·1667
0·0055	0·0054	−0·0001	0·0325	0·1692
0·0083	0·0084	+0·0001	0·0502	0·1653
0·0125	0·0124	−0·0001	0·0744	0·1680
0·0080	0·0083	+0·0003	0·0497	0·1610
0·0159	0·0159	±0·0000	0·0956	0·1663
0·0240	0·0241	+0·0001	0·1447	0·1657
0·0312	0·0308	−0·0004	0·1850	0·1687
0·0144	0·0147	+0·0003	0·0880	0·1636
0·0144	0·0145	+0·0001	0·0868	0·1659
0·0144	0·0141	−0·0003	0·0848	0·1698
0·0144	0·0140	−0·0004	0·0838	0·1719
0·0173	0·0172	−0·0001	0·1034	0·1673
0·0172	0·0172	±0·0000	0·1030	0·1670
0·0140	0·0142	+0·0002	0·0853	0·1642
0·0140	0·0140	±0·0000	0·0838	0·1670

Average factor: 0·1667.

It will be seen that there is a good agreement between the weights of citric acid taken and the weights found. The error in no case is as much as 0·5 mgrm. It is of interest to note that the factor 0·1667 corresponds exactly to one-sixth of the weight of the precipitate.

Experiments to try the smallest amount of citric acid that could be accurately determined by direct weighing gave the following results:

Weight of Citric Acid taken.	Weight of Citric Acid found.	Difference.	Weight of Precipitate.
0·0005	0·0008	+0·0003	0·0046
0·0011	0·0012	+0·0001	0·0070
0·0022	0·0021	−0·0001	0·0125

It will be seen that quantities less than 1 mgrm. give high results. Hence the limit must be placed at about this figure. Anything less than 3 mgrms. requires very careful working.

The experiments below were carried out to ascertain if the estimation could be made in the presence of other acids:

Acid.	Weight of Acid taken.	Citric Acid taken.	Citric Acid found.	Difference.	Weight of Precipitate.
Tartaric ...	0·0150	0·0144	0·0145	+ 0·0001	0·0872
" ...	0·0150	0·0172	0·0169	− 0·0003	0·1014
" ...	0·0300	0·0172	0·0167	− 0·0005	0·1004
Succinic ...	0·0150	0·0144	0·0143	− 0·0001	0·0858
" ...	0·0150	0·0140	0·0136	− 0·0004	0·0818
" ...	0·0300	0·0140	0·0137	− 0·0003	0·0822
Malic ...	0·0150	0·0143	0·0200	+ 0·0057	0·1202
" ...	0·0150	0·0144	0·0163	+ 0·0019	0·0976
" ...	0·0150	0·0156	0·0204	+ 0·0048	0·1223
Lactic ...	0·0150	0·0140	0·0155	+ 0·0015	0·0932
" ...	0·0240	0·0173	0·0215	+ 0·0042	0·1292
" ...	0·0480	0·0173	0·0296	+ 0·0122	0·1774
Oxalic ...	0·0150	0·0144	0·0142	− 0·0002	0·0850
Benzoic ...	0·0150	0·0140	0·0138	− 0·0002	0·0827
Salicylic ...	0·0150	0·0140	0·0173	+ 0·0033	0·1038
Phosphoric	0·0150	0·0140	0·0147	+ 0·0007	0·0880

In presence of tartaric, succinic, oxalic, benzoic, phosphoric, sulphuric, and acetic acids accurate results are obtained, but with malic, lactic, and salicylic acids the results are approximate.

Salicylic acid gave a salmon-coloured precipitate, which is probably a nitro compound; with tannic acid a voluminous orange-brown precipitate was obtained.

The percentage of mercury estimated by Volhard's ammonium sulphide method varied from about 72·5 to 74·0. The nitric acid determined by the phenolsulphonic acid and Kjeldahl methods was about 4 per cent. It is interesting to note that the precipitates obtained by Beau's modification of Denigès' method gave 72·5 per cent. of mercury and 3 to 4 per cent. of nitric acid. This seems to suggest that the precipitate is very similar, if not the same. These results do not correspond to any simple formula. The precipitates persistently retained traces of manganese, as shown by the colour method, and also possibly traces of uncombined mercury and nitric acid.

When heated, the precipitate suddenly decomposes. It is very soluble in hydrochloric acid, also in strong sulphuric acid and nitric acid. Drying seems to render the precipitate less soluble. Soluble haloid salts, as well as many other salts, readily dissolve it, this being the reason that it cannot be reprecipitated from a hydrochloric acid solution. When properly prepared, it is easily filtered and washed, and does not show any tendency to pass through the filter. It dries rapidly, and could probably be dried to a constant weight at 110° C. in three hours.

It has been found that three hours is about the minimum time for boiling, but if boiled much longer basic compounds tend to separate.

CONCLUSIONS.

1. It is shown that Beau's method is unreliable, due to the addition of hydrogen peroxide to remove manganese dioxide, and that no other oxidising agent can be

used. The manganese dioxide formed cannot be removed without oxidising or reducing the precipitate. In hot solutions, manganese dioxide slowly oxidises the precipitate.

2. The precipitate cannot be dissolved and reprecipitated. Halogen, phenylhydrazine, and other derivatives could not be prepared.

3. It has been found that a mixture of equal weights of mercuric nitrate, manganese nitrate, and nitric acid react with citric acid, yielding a precipitate after prolonged boiling which is exactly six times the weight of citric acid taken. The reaction has been studied, and a method for the accurate estimation of citric acid is given, together with details of the preparation of the reagent. The maximum error in the table is +0·0003 and −0·0004. Good results have been obtained in the presence of tartaric, succinic, and other organic acids, but malic and lactic acids gave high results.

In conclusion, I have to thank Mr. H. D. Richmond for the interest he has taken in the work and for allowing part of the early work to be done in his laboratory, also Mr. A. N. Gates, who has assisted me in the investigation.

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Estimation of Benzaldehyde and Oil of Bitter Almonds. F. D. Dodge. (*Eighth Int. Con. App. Chem.*, 1912, vol. 17, 15-20.)—In the estimation of benzaldehyde by the bisulphite method results sufficiently accurate for technical work may be obtained as follows: The aldehyde (0·15 grm.) is dissolved in 25 c.c. of $\frac{N}{5}$ sodium bisulphite solution, and the flask corked and allowed to stand for one and a half to two hours in ice-water, after which the liquid is titrated while still cold with $\frac{N}{10}$ iodine solution with starch as indicator. Simultaneously a blank estimation is made with 25 c.c. of the bisulphite solution, and the difference between the results calculated into benzaldehyde. One c.c. of iodine solution = 0·0053 grm. benzaldehyde. At best the results are only approximately correct, and only the free aldehyde is estimated in a natural oil. This also applies to the precipitation of the benzaldehyde as hydrazone, whilst in the oxime method of Bennett (ANALYST, 1909, **34**, 14) the presence of hydrocyanic acid apparently causes irregularities in the titration. Good results were obtained with artificial benzaldehyde by the use of a 2·5 N solution of potassium hydroxide in a method based on Cannizzaro's reaction:

$$2C_6H_5COH + KOH = C_6H_5COOK + C_6H_5.CH_2OH,$$

especially when the mixture was allowed to stand for twenty-four hours at the ordinary temperature instead of being heated before titrating the excess of alkali with standard acid. The objection to the method is that an error of only 0·05 c.c. of the alkali solution amounts to 1 to 2·5 per cent. of benzaldehyde. In the case of the natural oil the results were not satisfactory. A substance with a characteristic pleasant odour was isolated from the natural oil by removing the hydrocyanic acid by distillation with lime and ferrous sulphate, dissolving the residue in ether, precipitating aldehydes with sodium bisulphite, filtering off the crystals and washing them with ether. The ethereal extract when washed with sodium carbonate solution and evaporated *in vacuo* left about 0·2 per cent. of a heavy yellow oil, with an aroma which probably influences that of the oil. Under the same conditions artificial benzaldehyde yielded only traces of oil without characteristic odour. It is suggested that the presence of the new body may account for the superiority of the flavour of the natural oil. C. A. M.

Essential Oil of Cocoa. J. S. Bainbridge and S. H. Davies. (*Proc. Chem. Soc.*, 1912, **28**, 253.)—The authors have isolated, from 2,000 kilos of cocoa-nibs, 24 c.c. of a mixture of esters (formed during the fermentation of the cocoa-bean) with the true essential oil, which they regard as the aromatic principle of cocoa, and which consists chiefly of a *d*-linalol. Octoic and other fatty acids were also present in the oil, together with small quantities of an unidentified nitrogenous compound.

A. R. T.

Examination of Honey. G. Armani and J. Barboni. (*Ann. Lab. Chim. Cent. delle Gabelle*, 1912, **6**, 85-107.)—The presence of 10 per cent. of artificial honey may be detected by means of benzidine acetate with much more certainty than by the tests of Ley or Fiehe. The reagent is prepared by dissolving pure benzidine in sufficient dilute acetic acid to give a saturated solution. About 2 grms. of the honey are dissolved in about 10 c.c. of water and treated with 1 c.c. of the filtered reagent. In the presence of artificial honey a pronounced yellow coloration is produced, the intensity of which increases with the proportion of adulterant, so that it is possible to make an approximate colorimetric estimation of its quantity. This reaction is not due to the presence of nitrites, but is probably produced by oxymethyl-furfural in the artificial honey. Tables are given showing the analytical results and the colorations obtained in various tests in the analysis of a large number of samples of pure honey and mixtures of various origin. C. A. M.

Effect of Kiln-Drying at 140° F. on the Composition of the Hop. H. V. Tartar and B. Pilkington. (*J. Ind. and Eng. Chem.*, 1912, **4**, 839-840.)—The temperature at which hops are dried varies from about 70° to 140° F., the lower figure being more generally employed in Europe, and the higher in America. It is usually stated that at the higher temperatures soft resins are converted into hard, and valuable essential oils are lost. The authors, however, do not confirm this, and adduce many figures to show that the composition of the hop is almost identical whether samples are merely air-dried or dried at a temperature rising from 120° to 145° F. The resins were determined by methods previously described by the authors (ANALYST, 1912, **37**, 191) and Siller (*ibid.*, 1909, **34**, 485), tannin by Chapman's method (*ibid.*, 1908, **33**, 95; and 1909, **34**, 372), and wax by precipitating it from its solution in ether and weighing on a tared filter. Physical examination showed that the difference in the aroma of the air-dried and kiln-dried samples was hardly perceptible, different judges varying somewhat in their opinions. H. F. E. H.

Detection of Vegetable Oils in Margarine. L. Settim and L. Maurantonio. (*Ann. Lab. Chim. Cent. delle Gabelle*, 1912, **6**, 203-206.)—The greater proportion of the vegetable oils in margarine may be separated by heating 150 c.c. of the sample with about 200 c.c. of 95 per cent. alcohol with continual stirring, then allowing the mixture to cool, and decanting the alcoholic layer. The residue left on distillation of the alcohol will contain only minute quantities of "oleomargarine," and may be used in the special tests for cotton-seed oil, arachis oil, etc. C. A. M.

Estimation of Casein and Lactose in Milk. R. Malenfant. (*J. Pharm. Chim.*, 1912, **6**, 390-397.)—It is recommended that the casein be precipitated by means of alcohol acidified with acetic acid, the lactose being then estimated in the solution separated from the casein by filtration. Twenty-five c.c. of 65 per cent. alcohol and 3 drops of glacial acetic acid are placed in a flask, and 10 c.c. of the milk are added; the mixture is shaken for forty seconds, and, after the lapse of a further fifteen minutes, the coagulated casein is collected on a weighed filter and washed with 50 per cent. alcohol. The filtrate and washings are collected in a 100 c.c. flask, diluted with water to the mark, and the lactose is estimated in a portion of this solution by titration against Fehling's solution. The casein is now washed successively with boiling 95 per cent. alcohol, boiling acetone, and ether, then dried for seven hours at 100° C., and weighed. The casein thus obtained contains a quantity of insoluble salts, and the actual quantity of casein present is found by multiplying the weight by 0·925. W. P. S.

Interference of Hydrogen Peroxide with the Milk Tests for Formaldehyde. H. D. Gibbs. (*Philippine J. of Sci.*, 1912, **7**, 77-78.)—Both the Hehner and Leach tests for formaldehyde in milk fail in presence of hydrogen peroxide, though a positive result can still be obtained with the Rimini reaction. Mixtures of formaldehyde and hydrogen peroxide in milk were made with varying dilutions, the formaldehyde varying from 1 in 20,000 to 1 in 200,000, and the peroxide from 1 in 350 to 1 in 3,000, and in practically all cases only a brown, yellow, or reddish-brown colour was obtained, instead of the characteristic purple or violet. As showing that the failure of the tests is not entirely due to the reaction between hydrogen peroxide and formaldehyde before the application of the test, some of the mixtures which failed to give the characteristic colour were treated with sodium hydrogen sulphite and sulphur dioxide, and immediately tested with positive results. With even larger excesses of the peroxide this treatment was effective. A milk preservative called "Soldona," containing formaldehyde and hydrogen peroxide, is now on the market, presumably designed with a view to the interference above described.
H. F. E. H.

Total Solids of Milk. A. Splittgerber. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1912, **24**, 493-507.)—In the case of a complex material like the solids of milk, it is a matter of some difficulty to ascertain at what point of the drying process (as carried out in the usual way at a temperature of 100° C.) the water has been eliminated completely, as the continued loss in weight of the solids on prolonged drying is, in part, due to the decomposition of certain of the constituents. The author has therefore carried out a considerable number of experiments with the object of discovering the decompositions which milk constituents undergo when heated singly or together for many hours. The results obtained show that the alterations which take place in milk solids on prolonged heating are brought about almost entirely by lactic acid. Although this acid, when alone, volatilises fairly readily, it combines with certain milk constituents, such as casein, albumin, and lactose, and the greater part of the amount of the acid present becomes non-volatile unless the milk solids are heated for a considerable length of time, a portion of the acid being then liberated gradually and volatilised. This loss in weight, due to the volatilisation of lactic acid, does not take place to any appreciable extent during the first hour of the drying process, but after several hours it is plainly observable. At the same time, a drying period of one hour does not result in any marked decomposition of the various milk constituents. When heated for two hours or more, casein, mixtures of lactose with phosphates, and mixtures of casein with lactose and phosphates, undergo considerable alteration, whilst milk-fat, citric acid and its salts, albumin, and lactose, are scarcely, if at all, affected. It is also shown that when milk solids are dried until constant in weight, the weight found does not correspond with that of the sum of the constituents; the two weights agree more closely when the solids are dried for one hour at 100° C. The above-mentioned action of lactic acid affords an explanation of the low results which are obtained when the total solids of sour milk are estimated gravimetrically. W. P. S.

Detection of Saccharin in Vermouth. R. Guareschi. (*Ann. Lab. Chim. Cent. delle Gabelle*, 1912, **6**, 185-196.)—In most products saccharin may be detected by extraction with petroleum spirit after acidification with sulphuric acid, and tasting the white residue. In the case of vermouth, however, other substances which disguise the taste are also extracted. The following method is therefore recommended: From 300 to 500 c.c. of the sample are concentrated to about 150 to 200 c.c., acidified with 5 c.c. of dilute (1 : 3) phosphoric acid, and extracted with three successive portions of 150 c.c. of a mixture in equal parts of ether and petroleum spirit (boiling below 70° C.). The extract is washed and distilled, and the residue heated with a few c.c. of $\frac{N}{1}$ potassium permanganate solution to oxidise and destroy coumarin, vanillin, glycyrrhizic acid, etc., whilst leaving the saccharin unattacked. After complete oxidation the liquid is acidified with phosphoric acid and filtered. These operations are repeated two or three times, and finally, on evaporation of the liquid, a white residue will be left in which the saccharin may be identified by the usual reactions. C. A. M.

Methods for the Estimation of Saponin. M. Korsakoff. (*Compt. rend.*, 1912, **155**, 844-846.)—The method for the estimation of saponin established by Christophson is based on its precipitation in the form of a barium compound. The author has tested this method, hydrolysing the barium compound with sulphuric acid and titrating the dextrose produced. Purified saponin yields, on hydrolysis with 3 per cent. sulphuric acid at 105° C. for one hour, 60·16 per cent. of dextrose. On precipitating 2 grms. of saponin with baryta water, and hydrolysing the precipitate, only about 1·1 grms. of saponin were calculated from the dextrose produced, whilst a considerable further quantity was found in the mother-liquors. Moreover, when this method was applied to the extract of *Saponaria officinalis*, the saponin calculated from the dextrose obtained by the hydrolysis of the barium precipitate was nearly double that calculated from the sapogenin obtained. Hence, not only does the baryta method not precipitate the whole of the saponin, but from natural extracts it also precipitates bodies other than saponin which yield sugar on hydrolysis. A second method, proposed by Kruskal, is based on the formation of an unstable magnesium compound of saponin, the extraction of the glucoside from this by boiling alcohol, and its precipitation on cooling. Here, again, the author finds that only a portion of the saponin separates from a solution in 95 per cent. alcohol on cooling. The following improved method is now described: The plant to be analysed is dried and finely powdered; the powder is exhausted several times with boiling 60 per cent. alcohol. The alcoholic extract is filtered, the alcohol distilled off, and the residue evaporated on the water-bath with calcined magnesia. The magnesium compound is powdered and then extracted with boiling 80 per cent. alcohol; the liquid is filtered and the saponin precipitated by ether. The precipitate formed is dissolved in 3 per cent. sulphuric acid and hydrolysed for an hour in the autoclave at 105° C. The sapogenin produced is filtered off and washed until the washings are neutral; it is then dissolved in absolute alcohol, the alcohol is evaporated, and the weight of sapogenin is determined whence the equivalent of saponin may be calculated. J. F. B.

Iodine Titration Method for the Estimation of Tin, especially in Connection with the Estimation of Tin in Canned Foods. H. A. Baker. (*Eighth Int. Cong. App. Chem.*, 1912, vol. 18, 35-38.)—The tin sulphide obtained in the usual way from 100 grms. of the canned food (digestion with nitric and sulphuric acids, and precipitation with hydrogen sulphide) is dissolved in hydrochloric acid with the addition of potassium chlorate; a few pieces of aluminium foil are then added to the boiling solution to eliminate all the chlorine, and the tin is reduced to the metallic state by the addition of about 1 grm. of aluminium foil, this and subsequent operations being carried out while an atmosphere of carbon dioxide is maintained above the surface of the liquid. The mixture is again heated, when the tin dissolves to form stannous chloride, and, after the addition of a quantity of air-free water, the solution is titrated with $\frac{N}{100}$ iodine solution, using starch solution as indicator. Although the precipitate of tin sulphide may contain foreign substances such as lime, phosphorus, silica, lead, and even traces of iron, these will not interfere with the subsequent iodine titration. W. P. S.

Estimation of Higher Alcohols in Wine. T. von Fellenberg. (*Mitt. Lebensmittelunters. u. Hyg.*, 1912, **3**, 231-235; through *Chem. Zentralbl.*, 1912, II., 1851.)—The author has applied to wine the reaction which the higher alcohols give with salicylic aldehyde and concentrated sulphuric acid. Although the quantity of higher alcohols in wine gives no measure of its genuine character, the values obtained give some idea of the quantity of higher alcohols to be expected in the brandy perpared from it. For the estimation, 50 to 60 c.c. are distilled over from 100 c.c. of wine. The distillate is placed in a flask of 400 c.c. capacity, together with 0·5 c.c. of sulphuric acid (1 : 1); after five minutes it is made distinctly alkaline with sodium hydroxide, treated with 20 c.c. of $\frac{N}{10}$ silver nitrate solution, and heated for thirty minutes under a reflux condenser. Then about two-thirds are distilled off, and the distillate is made up to 100 c.c. Five c.c. of this distillate are treated in a 100 c.c. flask with 1 c.c. of a 1 per cent. solution of salicylic aldehyde and 4 c.c. of water. A standard is similarly made up with 5 c.c. of a solution containing 3 c.c. of higher alcohols (Rose's test) per 1,000 c.c. of absolute alcohol and diluted to contain the same amount of alcohol as the liquid under examination. To both flasks are added 20 c.c. of concentrated sulphuric acid, the flasks are shaken round, and after standing for forty-five minutes, 50 c.c. of sulphuric acid (1 : 1) are added; the two liquids are then compared in the colorimeter. Swiss wines, according to district, showed average values of 2·99 and 3·32 parts of higher alcohols per 1,000 of alcohol, from which it would appear that the official Swiss standard limit for cognacs of 1 per 1,000 is very low. J. F. B.

Colour Reaction of Wine. T. von Fellenberg. (*Mitt. Lebensmittelunters. u. Hyg.*, 1912, **3**, 228-231; through *Chem. Zentralbl.*, 1912, II., 1850-1851.)—Schaffer found a colour reaction with wine similar to that of phloroglucinol with vanillin and hydrochloric acid. According to the author, besides tannin, a constituent of the wine is concerned in it which has neither acid nor basic character, and is probably an unsaturated alcohol. This body gives with vanillin and hydrochloric acid a strong red coloration with a bluish tone, resembling acid-fuchsin. It was isolated by making the wine distinctly alkaline with sodium hydroxide and shaking out with ether (20 c.c. of ether for 10 c.c. of wine). The residue of the ether extract is treated with 2·5 c.c. of concentrated hydrochloric acid and a little vanillin, and is shortly afterwards diluted with 2·5 c.c. of 20 per cent. hydrochloric acid. After ten minutes the coloured solution is compared in a tube with a standard solution of acid-fuchsin (the latter substance dried at 100° C.), and the coloration is expressed in terms of mgrms. of acid-fuchsin per litre. With seven samples of Bernese wines the values ranged from 3·3 to 11·8. The reaction is of little value in the analysis of wines, but indicates the presence of an apparently new constituent of wines. J. F. B.

Estimation of Glycerol in Sweet Raisin Wines. B. Bardach and S. Silberstein. (*Chem. Zeit.*, 1912, **36**, 1401-1402.)—Although the usual method of estimating glycerol in wine yields only approximately correct results, it is employed very generally, and the quantity of glycerol found to be present in genuine wines is normal. In the case of sweet raisin wines, however, the amount of glycerol obtained by the method is abnormally high. This is due to the fact that raisins yield an aqueous infusion which, although free from alcohol, gives a considerable quantity of substance which counts as glycerol; this substance is not glycerol, but a mixture of nitrogenous organic matters with inorganic salts. The ratio of glycerol to alcohol in such wines is, therefore, usually found to be much too high, and the estimation is of little use, if any, as a criterion of the quality of the wines. W. P. S.

Investigations on Lead Precipitates from Wines; Estimation of Pentoses and Methylpentoses in Grapes and Wines. T. von Fellenberg. (*Mitt. Lebensmittelunters. u. Hyg.*, 1912, **3**, 213-227; through *Chem. Zentralbl.*, 1912, II., 1850.)—Natural wines contain considerable quantities of matters which are precipitated by basic lead acetate, but not by ordinary lead acetate, whilst these bodies are present in artificial wines only in small quantities. Basic lead acetate in excess tends to redissolve the precipitate, and basic lead nitrate is preferable. For its preparation, 400 c.c. of basic lead acetate solution are precipitated with 50 grms. of ammonium nitrate dissolved in a little water; the precipitate is washed with cold water, dissolved by heating with 3 litres of water and filtered; the excess of the salt separates on standing over-night and the mother-liquor serves as the reagent. For the estimation, 30 to 40 c.c. of wine are heated under a reflux condenser for five minutes with 0·5 grm. of animal charcoal, then neutralised carefully with 0·3 to 0·5 grm. of precipitated chalk, heated for one minute, cooled, and filtered. Preliminary tests are then made with lead acetate solution (190 grms. per litre) to ascertain what quantity of this reagent is required for precipitation, 2·5 c.c. of the wine filtrate are diluted with water to 5 c.c. and treated with increasing quantities of lead acetate, beginning with 0·15 c.c. The filtrates from these tests are tested until one is found which gives no further precipitate with a drop of lead acetate. Then 12·5 c.c. of the wine filtrate are treated with the ascertained proportion of lead acetate and diluted to 25 c.c. The liquid is filtered, and 10 c.c. of the filtrate (= 5 c.c. of wine) are treated in a tube of 40 c.c. capacity with 20 c.c. of the solution of basic lead nitrate, heated to 70° to 75° C. for five minutes and centrifuged for three to five minutes. After pouring off the clear liquid, the precipitate is stirred up with 7 c.c. of absolute alcohol, 7 c.c. of dry ether are added, and the mixture is again centrifuged. The liquid is poured off and the precipitate transferred with a little ether into one of the author's specially designed centrifugal tubes, in which it is centrifuged for three minutes at a speed of 1,200 revolutions, and its volume then read off. With twenty-four samples of natural wines the volumes ranged from 0·210 to 0·518 c.c.; grape marc wines showed only slightly lower values, but heavily diluted and fortified wines gave very small values. Examination of these basic lead nitrate precipitates showed that they contained succinic acid together with some sulphurous acid, inositol, pectin, pentosan, methylpentosan, and, for red wines, a trace of colouring matter. The pectin or parapectin of grapes contains methylpentose as well as pentose, but the quantity of these derived from the pectin is only a small portion of the total pentoses and methylpentoses. Grapes contain pentoses and methylpentoses both in the free state, not precipitated by basic lead nitrate, and in the form of pentosans and methylpentosans precipitated by that reagent. The grape-juice contains principally methylpentose, whilst the marc contains more pentose. Commercial dried-grape wines contain very little pentose and methylpentose; marc wines contain the two sugars in a different ratio from natural wines. J. F. B.

Detection and Estimation of Xanthin Bases in Cocoa, Tea, and Coffee, and their Preparations. S. Camilla and C. Patusi. (*Giorn. Farm. Chim.*, 1912, **61**, 337-385; through *Chem. Zentralbl.*, 1912, II., 1581.)—For the detection of small quantities of xanthin bases, the basic substance is isolated in any convenient manner, and treated with a few drops of very concentrated potassium hydroxide solution; saturated potassium permanganate solution is then gradually added. Reduction of the permanganate, with gas evolution, and the characteristic smell of carbylamine, show the presence of xanthin bases. To estimate theobromin and caffein, 10 grms. chocolate or cocoa are boiled for thirty minutes with 150 c.c. water and 50 c.c. $\frac{N}{1}$ sulphuric acid, made up to 500 c.c. and filtered hot; 250 c.c. of the cooled filtrate are neutralised with magnesium oxide, evaporated to 80 c.c. on the water-bath, and extracted for two to three hours with carbon tetrachloride in an apparatus described in the original paper. Caffein, a little theobromine, and fat, if any is present, pass into solution. The solution is evaporated to dryness with paraffin, the residue boiled several times with dilute sulphuric acid, the solution mixed with the liquid remaining in the extraction-apparatus, evaporated to dryness with 5 grms. magnesium oxide, and boiled up four or five times for ten minutes with separate quantities of chloroform of 100 c.c. each. The xanthin bases are obtained in sufficient purity on boiling off the chloroform. If the residue is coloured, it is dissolved in water and treated with potassium permanganate; the xanthin bases are then extracted from the pink solution with chloroform. The method has been successfully applied to tea, raw and roasted coffee, and coffee substitutes. O. E. M.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Experiments on Antigen and Antibody Actions: Differentiation of Proteins (Kidney, Liver, and Cancer Proteins). E. Rosenthal. (*Zeitsch. f. Immunitatsforsch. u. experim. Therap.*, 1912, **15**, 37-48; through *Chem. Zentralbl.*, 1912, II., 1848.)—By means of the epiphanin reaction it is possible to detect in guinea-pigs specific antibodies of the kidney protein by immunising with the kidney of another animal. This detection can also be made when the organism is induced to secrete antibodies by the breaking down and resorption of its own kidney parenchyma. Experiments carried out in accordance with these results yielded, however, a negative result when tried with guinea-pig's liver. According to this, the liver apparently possesses no antigen characters, or else the method, for some unknown reason, is not suitable for the detection of liver antibodies. Specific antibodies directed against tumour protein can be detected in guinea-pigs after previous immunisation with tumour material from the human subject or mice. Likewise in the serum from cancerous mice antibodies are present directed against mouse tumour protein, which apparently in a small degree also reacts towards human tumour antigen. J. F. B.

Estimation of Creatine, Creatinine, and Mono-amino Acids in Certain Fishes, Mollusca, and Crustacea. Y. Okuda. (*Eighth Int. Cong. App. Chem.*, 1912, vol. 18, 275-281.)—For the estimation of the creatine and creatinine, the finely-divided flesh was extracted with three successive quantities of water at

55° C., each extraction lasting one hour. The extracts were boiled, filtered, and the filtrate evaporated under reduced pressure to a small volume. The two bases were then estimated in this solution by Folin's method. A second portion of the flesh was extracted with water as above described. The soluble proteins were removed by coagulation and precipitation with phosphotungstic acid and sulphuric acid, and after the excess of the two latter had been removed by means of barium hydroxide, the monoamino acids were estimated by the formaldehyde titration method. The following results were obtained, the figures expressing grms. per 100 grms. of dry substance:

Kind of Fish, etc.	Creatine.	Creatinine.	Mono-amino Acid—Nitrogen.
Bonito	2·011	0·481	0·072
Tunnyfish	1·800	0·232	0·041
Salmon	1·525	0·182	—
Porgy	—	—	0·069
Snapper	3·327	0·308	—
Carp	2·020	0·369	0·194
Shark	3·242	0·663	—
Lobster	trace	trace	0·608
Crab	trace	trace	—
Cuttlefish	trace	trace	0·485
Clam	trace	trace	—

W. P. S.

Chemical and Bacteriological Study of Fresh and Frozen New Zealand Lamb and Mutton. A. M. Wright. (*J. Soc. Chem. Ind.*, 1912, **31**, 965-967.)—The changes in lamb and mutton when exposed to temperatures of 2° to 19° F. for varying periods up to one hundred and sixty days are described at length. The methods followed for the estimation of moisture, ash, fat, and total nitrogen, were those described in U.S. Department of Agriculture, Bureau of Chemistry, Bulletin 107, revised; the coagulable proteose, peptone, meat-base, and ammoniacal nitrogen, being determined as in the author's previous paper (ANALYST, 1912, **37**, 193).

Changes Due to Putrefaction.—Portions of finely-minced lamb and mutton were inoculated with infusions from putrefying meat, and examined at the end of two, four, seven, and fourteen days. The initial stages of putrefaction were marked by the transformation of insoluble into soluble proteins, and of coagulable proteins into proteoses, peptones, meat-bases, and ammonia. There was found to be a progressive increase in the amount of soluble matter, the total soluble nitrogen rising from 21·7 per cent. of that in the fresh meat to 75·5 per cent. after fourteen days. The most striking evidence of putrefactive change is given by the figures for ammoniacal nitrogen, which rise from 0·8 to 53 per cent. of the total nitrogen present. The changes due to ripening both in the presence and absence of bacterial infection were also studied, thymol and chloroform being used in the latter series. In the former case incipient decomposition commenced on the third day, while in presence of antiseptics enzymic changes ensued involving the increase of organic extractives, total soluble nitrogen, meat-base nitrogen, proteose and peptone nitrogen, for the first four to five days, after which no more change occurred.

Changes Due to Cold Storage.—There was a loss of moisture amounting to from 2·5 to 3·5 per cent., an increase in the amounts of proteose, peptone, and meat-base nitrogen, and a decrease in coagulable nitrogen, whilst ammoniacal nitrogen remained unchanged. These changes were similar to those occurring during the ripening of meat in absence of bacterial interference, the maximum change taking place in the lamb about the sixtieth day, and in the mutton between the ninetieth and one hundred and twentieth days.

The free acidity of the fat remained practically unchanged. The changes in chemical composition were probably due to enzyme action, and the meats remained in the same condition bacterially as when freshly killed. When the freezing and subsequent thawing were carried out slowly, there was no rupture or alteration of the structure of the tissues. H. F. E. H.

Estimation of Lipoids in Blood-Serum. L. Grimbert and M. Laudat. (*Compt. rend.*, 1912, **155**, 974-976.)—The authors propose the following simplified scheme, which has been proved to yield accurate results and afford valuable clinical evidence: Twenty c.c. of serum are treated with 100 c.c. of 95 per cent. alcohol to coagulate the proteins; after twelve hours' contact the precipitate is exhausted hot with a further quantity of alcohol in Kumagawa and Suto's apparatus. The alcoholic extracts are evaporated to a small bulk, and the residue, dried at 50° C., is exhausted with dry ether. The ethereal solution, when filtered through asbestos and evaporated, contains in the crude form the whole of the neutral fats, pre-existing fatty acids, phosphoric and other lipoids, and cholesterol. The extract is first saponified by heating for three hours with 50 c.c. of $\frac{N}{5}$ alcoholic potash per 0·2 grm. of extract. After evaporating the alcohol, the fatty acids are liberated by nitric acid and extracted by ether. The aqueous portion contains the phosphoric acid from the lipoids. The fatty acids are dried for an hour at 50° C., redissolved in dry ether; the solution is filtered through asbestos, evaporated, and the residue is dried in the oven for four or five hours to fix the pigments in an insoluble form. The residue is dissolved in light petroleum, the solution evaporated, dried at 50° C., and the mixture of fatty acids and cholesterol is weighed. This mixture is treated according to Kumagawa's method for the separation and estimation of the cholesterol. The liquid containing the phosphoric acid is evaporated in a large crucible on the water-bath, and calcined at a moderate heat to destroy the organic matter. The residue is extracted with a little very dilute nitric acid; the liquid is filtered, and collected in a centrifuge tube of 50 c.c. A large excess of molybdate reagent is added; the liquid is left at rest for two hours, then for four hours in an oven at 40° C., and centrifuged. The clear liquid is decanted off, and the precipitate is washed first with dilute molybdate and then with a little water. The precipitate is dried and weighed in the tube. The weight of phosphomolybdate divided by 2·3 gives the quantity of lipoids expressed in terms of distearo-lecithin. From the total fatty acids weighed above must be subtracted the oleic acid combined with the cholesterol, and the stearic acid combined with the lecithin. The factors are: Pure cholesterol, × 0·73; and lecithin, × 0·689. The residue represents the acids of the fats and the pre-existing fatty acids, which for all practical purposes may be taken together. Typical examples are given which illustrate the increased percentages of the above constituents in pathological sera as compared with normal. J. F. B.

Mett's Method for Determining the Activity of Pepsin and the Acidity-Maximum of Peptic Digestion. J. Christiansen. (*Biochem. Zeitsch.*, 1912, **46**, 257-287.)—As described originally by Mett (*Arch. Anat. Physiol.*, 1894, 68) the method consists in filling capillary tubes with egg albumin, coagulating the albumin by immersing the tubes for one minute in water at a temperature of 95° C., and then placing portions of these filled tubes in the pepsin solution to be tested, the latter being maintained at a definite temperature for twenty-four hours. At the end of this time the tubes are removed, and the extent to which the albumin has been dissolved from the tubes is measured and expressed in millimetres. The author finds that the method is quite suitable for comparing the activity of one pepsin with another, provided that certain precautions are taken in carrying out the test. In the first place, egg albumin varies in its behaviour towards pepsin, and the solubility is affected to a considerable extent according to the temperature at which the albumin has been coagulated. It is recommended that the tubes—about 40 cm. long, and having an internal diameter of about 1·5 mm. (the diameter, however, is of little consequence, but the measurement given is most convenient)—are filled with filtered albumin, and immersed in a large vessel filled with water at 85° C., and allowed to remain in the water until the latter has cooled to the ordinary temperature. When thus prepared, about 10 mm. of the albumin should be dissolved from both ends of a portion of the tube when placed in pepsin solution (0·3 per cent. of Armour's pepsin in $\frac{N}{10}$ hydrochloric acid) for twenty-four hours at 37° C. In practice it may be necessary to vary the initial temperature of coagulation a few degrees, so that different albumins may give this rate of solubility in pepsin solution. The stock tubes may be kept for six months without alteration if the ends of the tubes are sealed with shellac. The author finds that, for low concentrations of acid, the digestion is proportional to the time of contact, and that after some time the hydrochloric acid inhibits the action of pepsin; comparative tests should, therefore, not be prolonged for more than twenty-four hours. It is essential that the temperature during the digestion should be maintained as constant as possible, an alteration of a few degrees affecting the rate of digestion to a considerable extent.

In the case of human pepsin, the digestion proceeds most rapidly when the concentration of the hydrochloric acid in the solution is from 0·020 to 0·033 N, whilst for "animal" pepsin the maximum concentration lies between 0·050 and 0·075 N.

W. P. S.

Gold Bromide Test for Semen. A. de Dominicis. (*Vrtljschr. f. ger. Med. u. öffentl. Sanitätswesen*, 1912, [III.], **44**, 294, through *Chem. Zentralbl.*, 1912, II., 1789.)—A trace of fluid human semen is treated on a glass slip with concentrated gold bromide, and a cover-glass placed upon it; it is then carefully heated, and allowed to cool. With a magnification of 300 to 400, numerous garnet-coloured, cruciform, or square crystals, due to the presence of cholin, and also the elongated yellowish crystals produced by the semen may be seen. Fabrics are stretched upon glass slips, extracted with a few drops of distilled water, and the aqueous extract treated as above; if the semen (human) is not too fresh, numerous rectangular nut-brown crystals are obtained, the recognition of which by the reaction offers no difficulty. These crystals are due to the modification, with time, of the cholin; this can be shown by the extraction of human semen with alcohol, the residue obtained yielding, after a certain time, similar crystals. The modification of cholin which yields these crystals cannot be detected in any other manner. O. E. M.

Estimation of Skatol and Indol Together in Fæces. C. Moewes. (*Zeitsch. exper. Path. u. Ther.*, 1912, **11**, 555; through *Chem. Zentralbl.*, 1912, II., 1702.)—A mixture of the fæces with water in the proportion of 30 to 200 is steam-distilled with 1,000 c.c. of water, 200 c.c. of the distillate extracted with 50 c.c. of light petroleum, and 10 c.c. of the extract precipitated with 1 c.c. of a 2 per cent. solution of dimethylaminobenzaldehyde in 20 per cent. hydrochloric acid. The solution of the resulting dye in 4 c.c. of water is compared in a Plesch chromophotometer with a 1 in 100,000 test solution of equal parts skatol and indol. The results vary between 0·008 and 0·024 grm. skatol + indol per 100 grms. fæces.

O. E. M.

New Rapid Method for the Bacteriological Examination of Water and its Application for the Testing of Springs and Filter-Beds. P. T. Müller. (*Arch. Hyg.*, 1912, **75**, 189-223; through *Chem. Zentralbl.*, 1912, II., 1847.)—One hundred c.c. of the water to be examined are placed in a measuring cylinder with 5 c.c. of formalin and 5 drops of ferric chloride solution, mixed and allowed to settle for fifteen to thirty minutes. The clear liquid is poured off and the precipitate is treated with 5 drops of a concentrated alcoholic solution of gentian violet; it is then transferred to a specially-shaped centrifuge tube, and, after dipping for about a minute in the boiling water bath, it is centrifuged for a short time. 0·02 c.c. of the precipitate is transferred by means of a serological pipette to a microscope slide. The author employs a slide which has been etched in such a manner that a square of exactly 1 cm. remains clear. The precipitate is placed in the middle of this square and evenly spread by a platinum wire loop. The preparation is dried over a small flame, and, thus fixed, is covered with a drop of cedar-wood oil and examined without a cover-glass with an immersion-objective and the organisms counted. With waters containing 9,500 to 740,000 organisms per c.c., 99 per cent. of the organisms are precipitated by a single treatment with ferric chloride; highly infected waters should be suitably diluted before treatment. In testing spring waters the number of organisms found by this method was always higher than the number of colonies counted on plate cultures. When this method showed a water containing comparatively few organisms, cultivation on gelatin plates likewise showed only a few colonies. On the other hand, waters which showed only a few colonies on plate cultures showed sometimes fewer and sometimes far more organisms by the precipitation method. Deep bore waters were found both by plate cultures and by organism-counting to be nearly sterile. The method is applicable for the rapid control of the efficiency of sand filter-beds. J. F. B.

Yoghurt and Yoghurt Preparations. C. Griebel. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1912, **24**, 541-557.)—The author has examined various solid and liquid preparations which are sold for making Bulgarian or Turkish sour milk (Yoghurt); these preparations are supposed to contain the micro-organisms which, when cultivated in milk, convert the latter into Yoghurt. It is essential that the preparations or cultures should contain the *Bacillus bulgaricus* in an active condition, and the author describes the biological methods by which this bacillus may be identified together with the streptococci and diplococci which occur with it in true Yoghurt. Of sixteen liquid preparations examined, three were practically valueless, whilst only two out of six solid preparations were of any use; three of the latter did not contain *B. bulgaricus* in an active state. W. P. S.

ORGANIC ANALYSIS.

Detection of Cane-Sugar. S. Rothenfusser. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1912, **24**, 558-570.)—Results of further experiments on the detection of small quantities of cane-sugar in the presence of other sugars by the method described previously by the author (*cf.* ANALYST, 1912, **37**, 493) are recorded. Particular attention has been paid to the influence of other substances on the test. The following were found to have no interfering action: Alcohols and higher alcohols, aldehydes, organic acids, yeast constituents and decomposition products, organic esters, terpenes, preservatives, etc. The test may be applied to musts, dry wines, milk, honey, biscuits and infants' foods; in the case of certain sweet wines, however, some substance other than cane-sugar is present, and yields a positive reaction with the test. The nature of this substance has not been ascertained. Milk and materials which contain dextrins should be treated with acetone and filtered before the process is applied. W. P. S.

Estimation of Cyanogen Compounds in Gas Liquor. F. Weisser. (*Chem. Zeit.*, 1912, **36**, 1285-1287.)—Ammonium cyanide is estimated from the difference between total cyanogen and cyanogen existing as ammonium ferrocyanide. Total cyanogen is determined as follows: The gas liquor (500 c.c.) is shaken in a litre flask with 50 c.c. caustic potash solution (1 : 1) and 50 c.c. of a 10 per cent. solution of ferrous sulphate, and the flask is heated on the water-bath for least an hour. The contents of the flask are then cooled and made up to the mark, and the solution, in which all the cyanide now exists as ferrocyanide, is filtered from the precipitate of iron sulphide and hydroxide. An aliquot portion (100 c.c.) of the filtrate is made strongly acid with hydrochloric acid, and the ferrocyanogen precipitated as ferric ferrocyanide by addition of 5 c.c. of 10 per cent. ferric chloride solution. After twelve hours the precipitate is filtered off, washed with a little cold water, and decomposed by means of dilute caustic potash. After boiling, the solution of potassium ferrocyanide is filtered from the separated ferric hydroxide, acidified with sulphuric acid, and evaporated to dryness. The residue is gently ignited, taken up in dilute (1 : 1) sulphuric acid, and the iron is reduced with zinc and titrated with $\frac{N}{100}$ permanganate. One c.c. $\frac{N}{100}$ permanganate = 0·00156 grm. CN. Ferrocyanide is estimated by the following method, which depends on the assumption, said to be sound, that all the iron in filtered gas liquor exists as ammonium ferrocyanide: A measured portion of the filtered liquor is evaporated to dryness, and the residue is ignited and then dissolved in concentrated hydrochloric acid with addition of a few drops of nitric acid. Dilute (1 : 1) sulphuric acid is added, and the solution evaporated to fuming. After cooling and addition of more sulphuric acid, the iron is reduced by means of zinc, and titrated with permanganate. One c.c. $\frac{N}{100}$ permanganate = 0·00284 grm. $(NH_4)_4Fe(CN)_6$.

Thiocyanate is determined as follows: The gas liquor (50 c.c.) is diluted with an equal bulk of water and evaporated on the water-bath to about 30 c.c., to expel ammonia and hydrogen sulphide. The solution is made up to 100 c.c., and the thiocyanate in 20 c.c. is determined colorimetrically by addition of 10 c.c. of a 6 per cent. ferric chloride solution containing 10 per cent. of hydrochloric acid, followed by dilution to 500 c.c., filtration and comparison of 100 c.c. with a standard containing the same amount of free acid. As the intensity of colour is affected by the concentration of acid, the preliminary elimination of ammonia must not be omitted.

G. C. J.

Ethylene Dibromide as Cryoscopic Solvent. E. Moles. (*Zeitsch. f. physik. Chem.*, 1912, **80**, 531; through *Chem. Zentralbl.*, 1912, II., 1527.)—Ethylene dibromide is found to be as hygroscopic as nitrobenzene. The pure, perfectly dry substance melts at 9·975° C., and its cryoscopic constant is 12·5 (calculated, 12·35).

O. E. M.

Use of Hydrosulphite in Volumetric Analysis [of Dyestuffs]. I. W. Siegmund. (*Monatsh. f. Chem.*, 1911, **33**, 1431-1445.)—After investigating the various forms in which sodium hydrosulphite may be kept as a relatively stable solution suitable for use in volumetric analysis, the author has selected the following mixture as the best: One litre of water, 10 grms. of sodium hydrosulphite, concentrated B.A.S.F. powder, 15 c.c. of acetone, and 35 c.c. of ammonia, sp. gr. 0·91. The solution is stored in a three-necked Woulfe's bottle under a layer of paraffin oil, the bottle being permanently connected with a burette which can be filled by opening a cock. The liquid in the burette is protected from the air by a column of toluene. The titration flask has a cork with four holes: one of these carries a tube drawn out at the point and connected with the delivery end of the burette; through another passes a thermometer; whilst the remaining two serve for the passage of a current of carbon dioxide, generated in a Kipp's apparatus, through the flask, to expel the air before titration. The carbon dioxide is freed from oxygen by passing it through a washing bottle containing a solution of ferrous sulphate, Rochelle salt, and excess of ammonium carbonate. The titration flask stands in a water-bath which can be heated if necessary. For standardising the hydrosulphite, a solution of ferrous ammonium sulphate is employed which has been oxidised by permanganate and sulphuric acid; this solution should contain 2 grms. of iron per litre and the hydrosulphite should be restandardised before each series of determinations with 30 c.c. of the iron solution. The standardisation must be done in the cold, since the bisulphite developed would reduce the iron salt if heated; a few drops of thiocyanate are used as an indicator. The author has applied the method for the estimation of solutions of dyestuffs of the azo, nitro and triphenylmethane groups; the results agree well with the theoretical values for the pure compounds. The dyestuffs are dissolved in the proportion of about 2 grms. per litre, and 25, 50, or 100 c.c. of the solution are measured out into the titration flask. To the measured dyestuff solution are then added, for 50 c.c. of the dyestuff, 10 c.c. of a 20 per cent. solution of tartaric acid, and the mixture is boiled for a few minutes to expel the air. The flask is then placed in position in the water-bath and a current of carbon dioxide is passed through, whilst the temperature is adjusted at 70° to 80° C. Titration is effected at this temperature, the flask being agitated frequently and time being allowed for the hydrosulphite to exert its full effect. J. F. B.

Methoxyl Estimations with Hydriodic Acid in Presence of Phenol. F. Weishut. (*Monatsh. f. Chem.*, 1912, **33**, 1165-1172.)—By the condensation of the methylic ester of β-hydroxynaphthoic acid with anisic aldehyde, the author obtained a body which gave low results for methoxyl when treated by Zeisel's method, owing to its tendency to resinification. The addition of acetic anhydride, as recommended by Herzig for difficult cases of this nature, made very little improvement. It was found, however, that by performing the distillation in presence of phenol, the quantity of methyl iodide produced was in satisfactory agreement with the theoretical value. The author has made similar determinations of methoxyl groups in a range of other substances which fail to give satisfactory results by the ordinary method. Most of these substances only part with the whole of their methoxyl when heated for seven to fourteen hours with fuming hydriodic acid of sp. gr. 1·96. When treated by the author's method, with 2 to 3 c.c. of phenol in 10 c.c. of hydriodic acid of sp. gr. 1·7, most of these difficult substances gave perfectly satisfactory results in the normal time of one and a half hours. One of the substances tested—tetramethylellagic acid—required fuming hydriodic acid to produce the above result, even when phenol was used; whilst another—methyl nitroanisate—had to be heated with phenol and fuming hydriodic acid for five and a half hours before decomposition was complete. All the others (ten in number) behaved normally when phenol was employed. In carrying out this method there are placed in the distillation flask first the substance (about 0·2 grm.), then the melted phenol, and lastly the hydriodic acid. The author recommends that whenever normal values are not obtained by Zeisel's method in the ordinary way, the operation should be repeated with the modification above described. J. F. B.

Estimation of Total Fatty Acids in Cottonseed Foots. F. N. Smalley. (*Eighth Int. Cong. App. Chem.*, 1912, vol. 11, 31-35.)—The author recommends the following modification of the official method of the Inter-state Seed-Crushers' Association, which he finds to give more trustworthy results than any other method tried.

About 8 to 10 grms. of the well-mixed sample are saponified in a small extraction flask with alcoholic sodium hydroxide on a water-bath, the liquid being subsequently heated with occasional stirring until all alcohol is driven off. The soap is washed out into a beaker with about 400 c.c. of hot water, and digested on a water-bath till solution is complete. Dilute sulphuric acid is added in slight excess, and the heating continued on the water-bath till the layer of fatty acids has melted and become clear. The liquid is then cooled and filtered from the cake of fatty acids, and the acids washed with cold water till free from traces of sulphuric acid. The fatty acids are then allowed to dry by standing (preferably) overnight. The fatty acids on the filter-paper are transferred back to the original beaker, and the whole dissolved by the addition of 100 c.c. of petroleum-ether (boiling-point not exceeding 50° C.), boiling for a few minutes on the electric hot plate to assist solution. The solution is filtered through dry paper, and the residue treated with successive smaller portions of petroleum-ether till the fat is all dissolved. If only two such extractions are given, the residue in the filter-paper should be extracted in a Soxhlet apparatus. The solvent is evaporated and the residue of fatty acids heated to constant weight.

A. R. T.

Estimation of Total Fatty Acids in Cottonseed Foots. F. N. Smalley.

(*Eighth Int. Cong. App. Chem.*, 1912, vol. 11, 27-29.)—The following is recommended as a rapid factory method for the estimation of total fatty acids in foots, soap-stock, or similar materials, and consists in determining the iodine value, from which the fatty acids can be calculated by means of a factor.

Half a grm. of the well-mixed sample is treated with carbon tetrachloride (5 c.c.) and Wijs' iodine solution (20 c.c.) with the usual precautions. After complete solution of the material the mixture is allowed to stand for thirty minutes, when the titration is carried out with thiosulphate in the usual way. The iodine value of the sample, multiplied by 1·24, gives the percentage of fatty acids present.

The method yields good results when the soap-stock is not more than one week old; after this time, the iodine value falls, and low results for fatty acids are obtained by the use of the factor.

A. R. T.

Experiments on the "Cold Test" of Oil. A. Lowenstein and L. Beolio.

(*Eighth Int. Cong. App. Chem.*, 1912, vol. 11, 17-26.)—The "cold test" as employed for these experiments was substantially that outlined in Bulletin 99, p. 66, *Bureau Chem., U.S. Dept. Agriculture.* (Further information on the test is contained in Bulletin No. 105.) A pad of hair-felt was used to insulate the testing-bottle during the test.

To determine the effect of the presence of water in the oil, samples were prepared containing 0·05, 0·10, 0·20, and 0·30 per cent. of water, the oil and water being vigorously shaken until uniformly mixed. Forty c.c. of these oils were placed in duplicate 4-ounce oil bottles, and exposed to a temperature of -2° F. for eighteen hours. The results showed that the influence of water is in the direction of bringing the flowing-point of the oil towards the freezing-point of water. Tallow, having a high cold test (70° F.), gives a reduced reading proportionate to the water present; while in the case of neatsfoot oil, with a cold test below that of the freezing-point of water, the temperature is raised. The disturbances are small with small quantities only of water, and it would appear that samples should be tested as received unless excessive quantities of water or foreign solid impurities are present.

The next points tested were whether, where the oil is first frozen solid, the oil should be stirred while freezing or allowed to remain at rest, and whether the oil should stand for a definite time after freezing. An apparatus was devised to determine how much air was stirred into the oil during agitation, and from the results obtained the conclusion is drawn that concordant results for the cold test cannot be obtained unless an empirical method, rigidly followed out in all its details, is employed. After freezing, most oils should stand two hours (tallow oil, lard oil, etc.), though for certain types of oil one hour would be sufficient. The bath containing the oils for this test should have a temperature of 30° F. below the flowing-point of the oil.

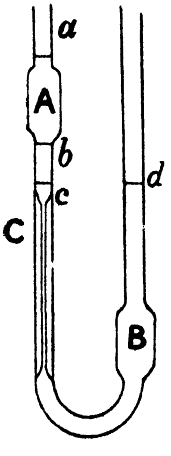
Experiments were also carried out in order to determine whether any different results were obtained in the "cloud test" (Bulletin 99, p. 66, *Bureau Chem., U.S. Dept. of Agriculture*) when a jacketed test-tube was employed instead of directly placing the oil in the bottle in contact with the freezing mixture. The air-jacketed tube method takes much time, and the results are practically identical with the simpler method. A. R. T.

Detection of Marine Animal Oil (Thran) in Other Oils and Fats. C. Stiepel. (*Seifensieder Zeit.*, 1912, **39**, 953-954; through *Chem. Zentralb.*, 1912, II., 1751.)—In criticism of Marcusson and Huber's modification of the insoluble bromide test (*Seifensieder Zeit.*, 1911, **38**, 249) for the detection of marine animal oils in other fats and oils, it is pointed out that the "hexabromide" value of linseed oil is considerably reduced, whilst the "octobromide" value of marine animal oil is eliminated by heating the oils. There is also a simultaneous decrease in the iodine value, which in the case of many marine animal oils falls considerably below 100. A negative result in the "octobromide" test cannot, therefore, of itself be regarded as conclusive of the absence of marine animal oil. For example, the new product *neutraline*, which has been reported by several chemists to consist of horse fat, or a mixture of that fat with hoof oils, has been found by the author to be a deodorised marine animal fat. C. A. M.

Detection of Mineral Oils in Oleins of Wool Fat. L. Settimj. (*Ann. Lab. Chim. Cent. delle Gabelle*, 1912, **6**, 197-201.)—The acetic anhydride method devised by Marcusson (*Mitt. König. Materialsprufungsamt*, 1904, **28**, 469) is shown to give good results only in the presence of considerable quantities of mineral oil. The following method is more sensitive and reliable: Ten c.c. of the wool fat olein are shaken with 40 c.c. of a mixture of 1 part of amyl alcohol and 2 parts of 96 per cent. ethyl alcohol in a 50 c.c. graduated stoppered cylinder. In the case of pure wool fat olein the liquid is clear at 20° C., whilst in the presence of 5 to 30 per cent. of mineral oil (Russian, Galician, Texan, etc.) the liquid remains turbid at 30° C., and on cooling deposits a layer of the mineral oil. The volume of this deposit is approximately 10 per cent. of the quantity of mineral oil in the sample. C. A. M.

Colour Reaction of Soya Bean Oil. L. Settimj. (*Ann. Lab. Chim. Cent. delle Gabelle*, 1912, **6**, 461-465.)—On shaking a mixture of 5 c.c. of soya bean oil with 2 c.c. of chloroform and 3 c.c. of a 2 per cent. aqueous solution of uranium nitrate, an intense lemon yellow emulsion is produced, whilst in the case of arachis, colza, cottonseed, maize, and sesame oils the emulsion is white. Olive oil usually gives a greenish emulsion, but some specimens give a slight yellowish tint similar to that produced by an oil adulterated with a small quantity of soya bean oil. The reaction with soya bean oil is sensitive, and the yellow coloration persists for several days. C. A. M.

Viscosity of Lubricating Oils. A. E. Dunstan and J. F. Strevens. (*J. Soc. Chem. Ind.*, 1912, **31**, 1063-1064.)—The authors advance the proposition that the viscosity of a lubricating oil can only be satisfactorily dealt with in the form of a temperature curve, so that the behaviour of an oil can be approximately predicted if the working conditions are known; and they further state that the viscosity of an oil should be expressed in absolute units of dynes per square cm. to allow of direct comparison.



Ostwald's modification of Poiseulle's viscometer is the instrument employed (see Fig.). Filtered oil is run into the viscometer to the marks *c*, *d*. The apparatus is suspended vertically, with a delicate thermometer, in a bath of paraffin wax or suitable clear high-flash oil, which is stirred by a glass paddle by means of a hot-air motor. The apparatus should be shielded from draughts of air. After the viscometer has been immersed fifteen minutes at a constant temperature, the levels of the oil are accurately adjusted to the marks *c*, *d*, using a warmed pipette to withdraw any oil, when a rubber tube is attached to the top of bulb *A*, and the oil sucked up above the mark *a*. It is then allowed to flow; and when the meniscus passes *a* the time (taken by a stop-watch) is noted for the flow between *a* and *b*. The average of four or five determinations is calculated. The dimensions of the apparatus are: Capillary, *C*, 6 cms. × 1 mm. bore; bulb *A*, 4 c.c.; bulb *B*, 8 c.c.; length over all, 15 cms. (made by Müller, Orme and Co., High Holborn). For lubricating oils it is advisable to take a series of readings between 70° C. and 200° C., and to plot a curve showing the variation of viscosity with the temperature.

For the calibration of the instrument, a very viscous liquid should be used as the standard. The viscosity of pure phenol, which is a suitable fluid, is as follows:

Temperature ° C.	Viscosity.
25	0·0850
45	0·0404
50	0·0337
60	0·0253
70	0·0197

If the time of flow of phenol in the viscometer was *t* seconds, then the viscosity = *K* × time × density of oil, where *K* is a constant for the particular instrument. *K* only varies slightly with the temperature. Example: Temperature ° C., 73·5; $t = 130·5$ seconds; log *K* for viscometer $= \bar{3}·0414$; sp. gr. of oil, 0·877; then viscosity $= K \times t \times d = 0·126$.

The results are of value as a means of obtaining the viscosity curve, and comparing this with the curve given by an oil of high lubricating value for a particular purpose.

The following are typical results:

Temperature ° C.	Ravison Rape Oil.	Standard Cylinder Oil.	Locomotive Cylinder Oil.	Crude American Unrefined Oil.
70	0·1460	0·9510		
80			0·6600	0·1620
100	0·0673	0·2630	0·2790	
115	—	—	—	0·0530
130	0·0327	—	—	
150	0·0219	0·0361	0·0574	0·0216
170	—	—	—	0·0139
180	—	—	0·0265	—
190		0·0237		
198		0·0187		

A. R. T.

Stearin of Wool Fat. E. Coen. (*Ann. Lab. Chim. Cent. delle Gabelle*, 1912, **6**, 567-579.)—The "stearin" of wool fat is obtained by pressing the distilled fat. Six samples examined by the author had a solidification-point above 48° C., and contained from 20 to 30 per cent. of solid unsaponifiable substances. The saponification value of the fats, excluding unsaponifiable matter, ranged from 157·9 to 168·5, with an average of 163; whilst the higher alcohols (expressed as cholesterol) in the unsaponifiable matter ranged from 18·7 to 27·3 per cent. (average 23 per cent.). The hydrocarbons differed from those in wool olein, consisting of a solid pasty mass with a somewhat high sp. gr. at 15° C. (0·924 to 0·936); a low iodine value (26 to 34); and a low specific optical rotation at 20° to 25° C. of +11·7° to +15·2°. In the case of four other samples the "stearin" melted below 48° C., contained less unsaponifiable matter (9 to 15 per cent.), and had saponification values ranging from 198·4 to 209·4. The hydrocarbons in the unsaponifiable matter (substances not combining with acetic anhydride) were liquid or semi-solid, and had an iodine value of 44·8 to 53·5, and an optical rotation at 20° to 25° C. of +12·1° to +21·2°. The presence of vaseline or paraffin wax in wool fat "stearin" would thus be indicated by the chemical and physical characters of these hydrocarbons. C. A. M.

Estimation of Paraffin Wax and Ceresin in their Mixtures. G. Armani and G. A. Rodano. (*Ann. Lab. Chim. Cent. delle Gabelle*, 1912, **6**, 109-118.)—Paraffin wax may be sharply distinguished from ceresin by the difference in their critical temperature of precipitation from a solution in a mixture of equal parts of absolute alcohol and benzene. Exactly 0·1 grm. of the sample is dissolved in 10 c.c.

sequent oxidation with bromine; (2) dry fusion with alkalis and an oxidising agent; (3) wet treatment with alkalis and oxidising agents; (4) Carius' method and modifications of it; (5) burning in oxygen at atmospheric pressure; (6) burning in a current of oxygen; (7) lamp methods; and (8) burning in a calorimetric bomb. The last method is applicable to all oils and combustible materials, is rapid and accurate, and is now used to the exclusion of all other methods in the laboratory of the Bureau of Mines. The application of lamp methods is restricted to oils which can be burned in a lamp, and Conradson (*Eighth Int. Cong. App. Chem.*, 1912, vol. 1, 133) has pointed out that the residue of unburned oil and the wick must be examined by a fusion method, as they retain sulphates and sulphonates. Methods (4) and (6) are accurate, but the latter requires much skilled attention, and the former is not free from danger to the operator. Method (5), due originally to Hempel, and advocated with modifications by Gräfe (*Zeitsch. angew. Chem.*, 1904, **17**, 616), is inaccurate, because combustion is not always complete, and methods (1), (2), and (3), though excellent for coal and coke, are not trustworthy for oils. G. C. J.

Wood Turpentines—their Analysis, Refining, and Composition. L. F. Hawley. (*Eighth Int. Cong. App. Chem.*, 1912, vol. 12, 41-100.—Five hundred grms. of the crude sample are distilled in a litre flask connected with a Hempel column dephlegmator (with thermometer) and a condenser. The distillation should be carried on at a maximum rate of 2 drops per second, and the distillate collected in fractions of 10 to 12 per cent., when the temperature is changing slowly, or of 4 to 5 per cent. when it is changing rapidly. As a rule, the whole of the turpentine substances will have passed over by the time a temperature of 180° C. (corr.) has been reached, just before the heavy oils begin to distil in quantity. Each fraction is weighed, and its boiling-point (corr.), sp. gr., and refractive index determined. In the last estimation a correction of 0·00047 for each degree by which the temperature of the room differs from 15° C. is applied. These several results are then preferably plotted in curves against the total percentage weight of the fractions. Typical results obtained in the distillation of samples of old gum turpentine are shown in the following table:

Temperature corr. to 760 mm. ° C.	Percentage Weight.		Sp. Gr. at 15° C.	Refractive Index at 15°.
	Single Fractions.	Combined Fractions.		
158·5	9·5	9·5	0·8650	1·4714
159·0	11·0	20·5	0·8651	1·4715
159·3	11·0	31·5	0·8653	1·4716
159·7	12·2	43·7	0·8655	1·4720
160·0	11·0	54·7	0·8656	1·4723
160·8	12·8	67·5	0·8660	1·4727
161·7	10·0	77·5	0·8665	1·4734
164·5	10·5	88·0	0·8675	1·4750
169·5	6·8	94·8	0·8689	1·4780
Residue	5·0	99·8	0·9450	1·4943

In the case of a wood turpentine, the first 70 to 75 per cent. might have consisted of a mixture of the same terpenes as those of gum turpentine mentioned above, the somewhat lower physical characters being attributable to the presence of smaller proportions of nopinene or camphene, or both. But between the points of the distillation of 75 and 95 per cent. the sp. gr. of the distillate decreased, while the refractive index increased. This indicated the presence of a substance of higher boiling-point and lower sp. gr. than "pinene" (which here includes also camphene and nopinene). The terpene dipentene (b.-pt. 175° C.; sp. gr. 0·849 at 15° C.; $[N]_{D\,15^{\circ}\,C.} = 1{\cdot}471$) was isolated from this sample, and, in the author's opinion, a decrease in the sp. gr. as the distillation temperature approaches 175° C. is probably a good indication of the presence of this terpene. The separation of the heavy oils from the dipentene was quite sharp, and the conclusion was drawn that this wood turpentine contained about 95 per cent. of terpenes, containing about 80 per cent. of "pinene" and 15 per cent. of dipentene, together with 5 per cent. of heavy oils of high b.-pt., probably non-terpenes. Special experiments showed that there was only a slight change in the composition of wood turpentine, whether by direct distillation or in a current of steam. Towards the very end of the distillation a slight polymerisation of the terpenes to substances of higher physical characters apparently occurred. The main differences between gum and wood turpentines are that the latter usually contain heavy oils, which are generally absent from the former. Even in the case of an old gum turpentine containing heavy oils, such as that described above, it is probable that these oils are of different composition. No dipentene has been found in seventy-five samples of gum turpentine examined. Its presence in wood turpentine may be due to the volatile oil in the original wood, or to transformation of some of the "pinene" by the high temperatures of distillation. Wood turpentines also usually contain light oils, for their removal during refining is difficult and requires a fractionating column. It is also probable that "rosin spirits" are present in wood turpentine, and account for the odour and colour of the fractions, though they do not materially affect the distillation curves. With regard to the technical qualities, the volatility of a sample would be affected by large amounts of heavy oil, but would probably not be much decreased by proportions up to 10 per cent. The solvent action and drying properties of dipentene appear to be equal to those of "pinene," and the small proportions of other ingredients in wood turpentine probably do not have much influence. Experiments on samples of refined wood turpentine showed that no great change in composition takes place on storage.

C. A. M.

INORGANIC ANALYSIS.

Acidity and Alkalinity of Natural Waters. J. Walker and S. A. Kay. (*J. Soc. Chem. Ind.*, 1912, **31**, 1013-1016.)—A practical application of Sörensen's method (*Biochem. Zeitsch.*, 1909, **21**, 131; **22**, 352) for the determination of hydrion concentrations. A new and more convenient notation is proposed with the hydrion (or hydroxidion) concentration of pure water at the same temperature as unity. Thus, a water with a hydrion concentration of $1{\cdot}36 \times 10^{7}$ at 18° C. is described as having a *relative acidity* of 2, or a *relative alkalinity* of 0·5, since the hydrion (or hydroxidion) concentration of pure water at 18° C. is $0{\cdot}68 \times 10^{-7}$. An advantage of the method is that, whereas the hydrion concentration varies considerably with small changes of temperature, the *relative acidity* or *alkalinity*, as defined above, is nearly independent of the temperature at which the comparison is made. The method, therefore, is analogous to, and shares the practical advantages of, that by which the specific gravities of dilute aqueous solutions are determined by comparison of their densities with that of water at the same temperature—not necessarily a standard temperature.

The following solutions are required: (1) A $\frac{N}{15}$ solution of $Na_2HPO_4.12H_2O$, made by dissolving 23·88 grms. of clear crystals picked from a supply of the "chemically pure" substance, and diluting to 1,000 c.c.; (2) a $\frac{N}{15}$ solution of KH_2PO_4, made by dissolving 9·08 grms. of the pure salt (specially recrystallised and dried at 100° C.), and diluting to 1,000 c.c.; (3) a mixture of (1) and (2) in the ratio 612 : 388; and (4) a 0·1 per cent. solution of azolitmin (Kahlbaum). The solution (3) is neutral.

Of the water to be examined, 25 c.c. is tinted with a measured quantity of the azolitmin solution (1 c.c. is recommended as a convenient amount), and the tint compared with that of 25 c.c. of the neutral solution (3), to which an equal amount of azolitmin is added. If the sample proves to be decidedly acid, 25 c.c. of the acid phosphate (2) is measured out into a similar basin or Nessler cylinder, azolitmin is added, and alkaline phosphate (1) run in from a burette until a match is obtained, more of the sample being added to the other basin or cylinder from time to time to keep the concentration of the indicator in the two vessels approximately the same. In a second titration it is easy to secure almost exact equality of bulk in the two vessels. If the water sample is coloured, resort should be had to the compensation method of Walpole (*Proc. Physiol. Soc.*, 1910, **40**, 27) in the second titration. The acidity of the sample, corresponding with the composition of the phosphate mixture at the matching-point is given in Table I.:

TABLE I.—FOR DECIDEDLY ACID WATERS.

Volume of KH_2PO_4 used = 25 c.c.

Acidity.	Volume of Na_2HPO_4 required.	Acidity.	Volume of Na_2HPO_4 required.
300·0	—	4·5	8·07
100·0	0·28	4·0	9·15
50·0	0·63	3·5	10·5
20·0	1·68	3·0	12·3
15·0	2·23	2·5	15·2
10·0	3·57	2·0	19·2
9·0	3·97	1·8	21·4
8·0	4·52	1·6	24·2
7·0	5·18	1·4	27·7
6·0	6·05	1·2	32·7
5·0	7·26	(Neutral) 1·0	39·4

If the sample is alkaline or nearly neutral, 25 c.c. of the alkaline phosphate is measured out, the indicator is added, and the acid phosphate is run in until a good match is obtained. The acidity or alkalinity of the sample, corresponding with the volume of acid phosphate required, is given in Table II. :

TABLE II.—FOR NEARLY NEUTRAL AND ALKALINE WATERS.

Volume of Na_2HPO_4 used = 25 c.c.

Acidity.	Alkalinity.	Volume of KH_2PO_4 required.	Acidity.	Alkalinity.	Volume of KH_2PO_4 required.
—	20·0	—	—	3·0	4·52
—	15·0	0·24	—	2·5	5·56
—	10·0	0·75	—	2·0	7·22
—	9·0	0·91	—	1·8	8·15
—	8·0	1·11	—	1·6	9·30
—	7·0	1·39	—	1·4	10·8
—	6·0	1·77	—	1·2	12·9
—	5·0	2·35	(Neutral) 1·0	1·0	15·8
—	4·5	2·72	1·2	—	19·1
—	4·0	3·15	1·4	—	22·6
—	3·5	3·74	1·6	—	25·8

If many samples, differing only slightly in acidity or alkalinity, have to be examined, it is convenient to prepare a series of standards covering the probable range of the samples. The paper includes a table, by reference to which such standards can readily be prepared in quantity by mixing solutions (1) and (2).

If the standards are diluted with water, their relative acidity is increased, but the error introduced by the use of wet vessels is negligible. Cooling increases the acidity, but the separate effects of temperature on the standards and the sample almost exactly counterbalance each other. The temperature of the standards and the sample should be identical, but a difference between them of 5° C. only introduces an error of 5 per cent., or about 0·05 of a unit in the neighbourhood of the neutral point. The phosphate solutions remain unchanged in glass bottles for many months at least.

The importance of the method described depends on the fact that it affords a measure of the actual acidity or alkalinity of a water, and not merely the potential acidity or alkalinity as given by ordinary titration. It is on this actual state of the water that its rate of action on metals depends. An example is quoted of a water which was rendered dangerously active towards lead by treatment with aluminium sulphate, though the amount of the latter used was equivalent to less than half the bicarbonate hardness of the water, which after treatment still possessed temporary hardness, and no free acid save carbonic acid, to excess of which its high relative acidity was due.

G. C. J.

Detection of Antimony in Alloys. R. Belasio. (*Ann. Lab. Chim. Cent. delle Gabelle*, 1912, **6**, 501-503.)—From 0·5 to 1 grm. of the finely-divided alloy is treated with 2 c.c. of boiling concentrated sulphuric acid, and the mixture cooled, diluted with an equal volume of water, cooled and filtered from the insoluble lead sulphate. A few drops of the filtrate are heated in a porcelain basin with a crystal of sodium sulphite until the liberated sulphur dioxide has been expelled, after which

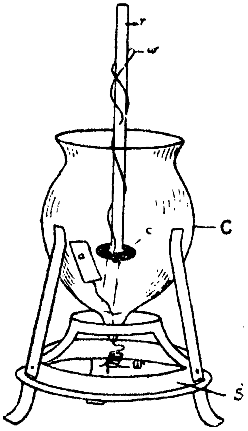
1 or 2 drops of a solution of 1 grm. of potassium iodide and 1 grm. of cæsium chloride in 5 c.c. of water are added. In the presence of antimony a stable rose-coloured precipitate of iodide of cæsium and antimony is obtained. Under the same conditions bismuth gives a fugitive yellow coloration and a brown precipitate, whilst copper gives a light yellow precipitate. C. A. M.

Cadmium Nitrate in Quantitative Analysis. A. Vorisek. (*Eighth Int. Cong. App. Chem.*, 1912, vol. 17, 91-102.)—A solution of cadmium nitrate may be used to separate ferrocyanides, ferricyanides, and sulphides, from the other anions precipitated as silver salts (chloride, bromide, iodide, cyanide, and thiocyanate). The solution of the salts is neutralised, an excess of 10 per cent. cadmium nitrate solution added, and potassium hydroxide solution run in until the reaction is faintly alkaline, after which the excess of alkali is neutralised with a few drops of the cadmium solution or of dilute acetic acid, and the mixture diluted, heated on the water-bath for five to ten minutes, and filtered while hot. When ferricyanide is present, traces may pass into the filtrate, and to remove these it is necessary to boil the filtrate with a few drops of sulphurous acid solution and filter it again. Portions of the filtrate are then tested for cyanide (about four-fifths of which will remain in solution), and for chloride, bromide, and iodide. Thiocyanate is eliminated by means of mercuric chloride solution.

Precipitation of Tartaric and Citric Acids by Cadmium.—A solution of cadmium nitrate in the presence of potassium hydroxide completely precipitates tartaric and citric acid from neutral solutions, the excess of alkali being neutralised as described above. The precipitate is filtered while hot, washed, and stirred with cold 5 per cent. acetic acid until disintegrated, after which the liquid is filtered and the solution of cadmium citrate and tartrate is evaporated nearly to dryness. The residue is taken up with water, the cadmium separated by means of hydrogen sulphide, and the filtrate evaporated to obtain the free citric and tartaric acid. C. A. M.

Sensitive Reaction for Molybdic Acid. P. Melikow. (*J. Russ. Phys.-Chem. Soc.*, 1912, **44**, 608; through *Chem. Zentralbl.*, 1912, II., 1579.)—The residue obtained by evaporating the solution under examination on the water-bath is rendered alkaline with concentrated ammonia solution, and a 3 to 4 per cent. solution of hydrogen peroxide added; the presence of molybdic acid in the original solution is shown by a pink or red coloration due to ammonium permolybdate. If the liquid is then concentrated, and sulphuric or nitric acid added, a yellow solution of permolybdic acid is obtained. O. E. M.

Electro-Analysis with Light Platinum Electrodes. F. A. Gooch and W. L. Burdick. (*Zeitsch. anorg. Chem.*, 1912, **78**, 213-220.)—The total weight of platinum in the apparatus illustrated does not exceed 1 grm. The glass cell, of about 35 c.c. capacity, is constructed from the top of a thistle funnel, and rests in a brass spring support. A similar support of aluminium is used to support the cell on the balance pan. [In use the cell is pressed down into the stand as far as it will go, but is not so figured, as parts of the apparatus would then be obscured.] The anode *a* is a piece of thin platinum-foil about 1 cm. square, welded to a platinum wire, *w′*, which penetrates the wall of the cell. The cathode *c* consists of a disc of gauze or foil, also about 1 square cm. in area, which is fused to the glass rod *r*. The wire *w″* makes connection between *c* and the source of current. As the use of such small electrodes implies a high current density, unless the electrolysis is to be a prolonged operation, the apparatus has been designed to deal with deposits with no pretence to be adherent. An essential part of the equipment, therefore, is a filter-tube, which is not figured, but which consists of a short piece of tubing to one end of which a disc of platinum-foil is fused. Before attaching the foil it is pierced with pinholes, and the burred side is turned away from the glass. Before commencing an analysis, this filter-tube is plunged into some asbestos pulp, its open end is connected to a pump by means of rubber tube, and the filter-tube thus prepared is dried and weighed together with the cell and the cathode with its connecting wire and glass rod support. The liquid to be electrolysed (about 15 c.c.) is next introduced into the cell, which is covered with an inverted funnel or split watch-glass, and the cathode is connected to gear which gives it a rotation of about 300 r.p.m. When electrolysis is complete the filter-tube is dipped into the electrolyte, its open end being connected to the pump, and the cell is repeatedly filled up with water, and finally with alcohol. The cell, cathode, and filter-tube, are then dried in an air-bath, and finally weighed. Several results with copper and nickel are given. With a current of about 1 amp. at 5 to 6 volts ($N.D._{100} = 50$ to 60), 0·1 grm. of either metal may be deposited in twenty to thirty minutes with an error not exceeding ± 0·5 mgrm. G. C. J.



Determination of Lead Sulphate and the Ultimate Estimation of "Sublimed White Lead" in Rubber. J. A. Schaeffer.

(*J. Ind. Eng. Chem.*, 1912, **4**, 836-838.)—"Sublimed white lead," as placed on the market, is a basic sulphate of lead of the average percentage composition: $PbSO_4$, 78·5; PbO, 16·0; ZnO, 5·5. Commercial samples differ very little in percentage composition. The properties of the material, which consists of very finely-divided amorphous particles, are said to recommend it as a constituent of rubber mixings. Its use is limited, however, in cases where specifications set a limit to the total sulphur content. There is reason to believe that its more extended use would be tolerated if ready means existed for differentiating between inert sulphur, existing as lead sulphate, and active sulphur, to which purchasers are anxious to set a limit. The following method has, therefore, been devised for estimating lead sulphate in rubber: The sample (2 grms.) is placed in the middle of a hard glass tube, about 8 inches by 1 inch, and gently heated, whilst a stream of carbon dioxide is passed through the tube. When the organic matter is all driven off, the dry residue is ground in a small agate mortar, transferred to a flask, which is filled with carbon dioxide, and decomposed by boiling with 25 c.c. of hydrochloric acid, a current of carbon dioxide being passed all the while to avoid risk of oxidising sulphide to sulphate. Twenty minutes' boiling suffices to eliminate hydrogen sulphide and to bring nearly the whole of the lead sulphate into solution. The solution is diluted and filtered, and any residue is washed with hot water, ignited in a porcelain crucible, digested with hydrochloric acid, and the solution filtered and added to the principal solution. Lead sulphate equivalent to 0·1 to 0·2 per cent. on the original rubber may be recovered by this treatment of the "insoluble" residue. The combined filtrates are nearly neutralised with ammonia, neutralisation is completed by the cautious addition of sodium carbonate, and an excess of about 2 grms. sodium carbonate is then added. The solution is boiled for fifteen minutes, filtered, and the precipitate is washed. The sulphate in the filtrate and washings is then determined as barium sulphate, and calculated to sulphur and lead sulphate. Test analyses show that oxidation of sulphide to sulphate is not wholly prevented, that sulphate sulphur equivalent to 0·1 per cent. may be found when none was originally present, and that 2·1 per cent. may be found when only 2·0 per cent. (equivalent to 19 per cent. of lead sulphate) was present in the original sample. These numbers illustrate the extreme error of the method, which never gives results below the truth. Methods for the estimation of lead sulphate in rubber mixings which also contain calcium and barium sulphate will be dealt with in another paper. G. C. J.

Detection of Nickel in Plated Articles. G. Silvestri and A. Cappelli. (*Ann. Lab. Chim. Cent. delle Gabelle*, 1912, **6**, 119-120.)—Reichard's methylamine hydrochloride test for nickel may be used for the detection of that metal in plated objects. If a fragment of nicked-plated iron is heated with a drop of hydrochloric acid and a crystal of methylamine hydrochloride, a blue coloration is produced, which disappears on cooling. Cobalt under the same conditions gives an azure blue coloration, but, unlike the nickel coloration, this persists after cooling. C. A. M.

New Gasometric Method for the Estimation of Nitric Oxide: I. O. Baudisch and G. Klinger. (*Ber.*, 1912, **45**, 3231-3236.)—The new method for the estimation of nitric oxide in gas analysis depends on its oxidation by means of air, with the formation of nitrogen trioxide and the absorption of the latter in the form of nitrite by means of potassium hydroxide. Raschig has studied the oxidation of nitric oxide, and has found that it takes place in two stages. The first phase corresponds to the formation of the trioxide, and takes place with extreme rapidity; the second phase corresponds to the formation of the tetroxide, and requires an appreciable time for its completion. In order to limit the reaction to the first phase, it is only necessary to absorb the trioxide as fast as it is produced, so that an excess of oxygen cannot come into play. The reaction is then expressed by the equation

$$\begin{array}{ll} 4NO + O_2 + 4KOH & = 4KNO_2 + 2H_2O. \\ 4 \text{ vols.} + 1 \text{ vol.} & = 0 \text{ vol.} \end{array}$$

The operation is performed in a Fuller's gas pipette into which a few sticks of moist potassium hydroxide are inserted and all the air is then expelled by mercury. A measured volume of the gas mixture to be analysed is introduced so that it surrounds the potash; next, a measured volume of air in excess is admitted, and the reaction allowed to take place. The residual gas is withdrawn and measured in the burette. Four-fifths of the observed contraction are due to the nitric oxide. The air used should previously be freed from carbon dioxide. The new method is perfectly accurate and very rapid, and the presence of other gases, such as nitrous oxide or hydrogen, which interfere with other methods, is quite immaterial. When the oxidation is performed in the reverse manner by admitting the nitric oxide to the excess of air standing in presence of potash, the results are inaccurate, because a mixture of trioxide and tetroxide is produced, with the absorption of a variable quantity of oxygen. J. F. B.

Determination of Phosphorus in Commercial Acetylene. L. M. Dennis and W. J. O'Brien. (*J. Ind. Eng. Chem.*, 1912, **4**, 834-836.)—The paper deals really with the examination of commercial carbide rather than acetylene. For the estimation of calcium phosphide, the authors recommend a modification of the method of Lunge and Cedercreutz, which depends on the generation of phosphine and its subsequent oxidation, by means of hypochlorite, to phosphoric acid, which is finally determined as magnesium pyrophosphate. A Kipp apparatus with 4-inch bulbs is used for the decomposition of the carbide, and its gas outlet is connected to two Friedrichs' gas-washing bottles, arranged in series, and each charged with about 75 c.c. of a solution containing approximately 3 per cent. of actual NaClO. Before introducing the sample, sufficient brine is introduced into the Kipp apparatus to seal the end of the stem attached to the upper bulb. The apparatus is next connected to the gas-washing bottles and air swept out of the whole series by introducing hydrogen through the upper tubulure of the Kipp apparatus. The gas-washing bottles are temporarily disconnected, 50 grms. of carbide are introduced into the middle bulb of the Kipp apparatus, connection is again made with the gas-washing apparatus, and hydrogen is passed for a further five minutes. Sufficient brine is then introduced through the upper tubulure of the Kipp apparatus to enable the apparatus to function as a gas generator, and the cock between the apparatus and the gas-washing bottles is so adjustsd that bubbles rise through the latter at a rate slightly faster than can be counted. Two hours serve for the decomposition of 50 grms. of carbide. When decomposition is complete, the acetylene remaining in the generator is driven through the absorption bottles by means of a current of hydrogen. The contents of the gas-washing bottles are transferred to a beaker, acidified with 10 c.c. of hydrochloric acid, boiled to expel chlorine, and rendered alkaline with ammonia, and the phosphate present is then precipitated with magnesia mixture. Carbide made from carefully-selected materials yields less than 10 mgrms. of magnesium pyrophosphate when tested in this way. Small quantities like this may be estimated with an error of ±0·2 per cent., whilst the error attaching to determinations of quantities five times as large is about ±1 per cent. It is usual to state results as c.c. phosphine per 100 c.c. acetylene. For this purpose it is necessary to determine the total volume of gas liberated by a definite weight of the carbide, and the most convenient method is that which depends on the loss in weight of an apparatus in which a weighed quantity of carbide is decomposed by brine, the apparatus including a calcium chloride tube, to deprive the escaping acetylene of moisture.

G. C. J.

Estimation of Potassium in Fertilisers, Soil Extracts, and Plant Ashes. W. A. Davis. (*J. Agric. Sci.,* 1912, **5**, 52-66.)—The platinum chloride method of estimating potassium is in many respects unsatisfactory, and has been largely superseded, especially in Germany, by the perchloric acid method of Wense and Caspari (*cf.* ANALYST, 1910, **35**, 417). To obtain the best results, the perchlorate precipitate should be washed with 95 per cent. alcohol saturated with potassium perchlorate at the washing temperature. The presence of barium, magnesium and calcium chlorides and sodium phosphate are all shown experimentally to be without harmful effect, and these salts need not be removed. Potassium sulphate can be estimated direct, using a sufficient excess of perchloric acid without conversion into chloride by means of barium chloride; if, however, the sulphate is converted into chloride by the Stassfurt method, the *exact* precipitation of the sulphate is not imperative as with the platinum method. All uncertainty as to the atomic weight of platinum is eliminated by the use of the perchloric method. Full details as to the preparation and treatment of the precipitate are given, for which reference should be made to the original paper.

H. F. E. H.

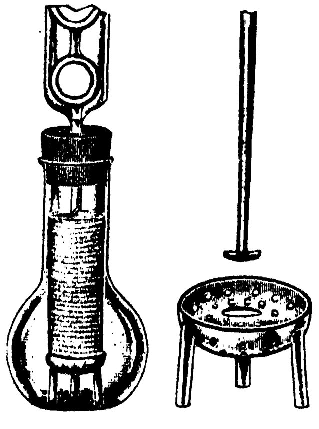
Estimation of Water in Silicate Minerals and Rocks. M. Dittrich. (*Zeitsch. anorg. Chem.*, 1912, **78**, 191-200.)—Penfield's method may give results far below the truth unless the tube be wrapped round with platinum as Penfield directed, and the temperature raised to such a point that the platinum adheres to the glass. Many minerals and rocks fail to give up the whole of their water when heated alone in quartz or platin-iridium tubes, if the highest temperature employed is that of an ordinary blowpipe flame. If an electric furnace capable of giving a temperature of 1250° C. be available, heating alone in a quartz tube, with subsequent direct weighing of the water evolved, gives results agreeing with those obtained by the method of Dittrich and Eitel (ANALYST, 1912, **37**, 380). The former method has the advantage that a determination of carbon dioxide may be combined with it. On the other hand, it is not available in presence of fluorides, whereas the method of Dittrich and Eitel is, and, moreover, gives a residue which is immediately available for the determination of silica, alumina, etc. The criticism of Penfield's and other methods is supported by test numbers.

G. C. J.

Gravimetric Estimation of Zinc. H. Schilling. (*Chem. Zeit.*, 1912, **36**, 1352.)—It is proposed to substitute benzene-sulphonic acid for acetic acid in the precipitation of zinc as sulphide. The advantages of the modification are the more rapid subsidence of the precipitate, its more granular condition which determines more rapid filtration, and the fact that it exhibits no tendency to pass through the filter when washed with distilled water. The solution is freed in the usual manner from silica and from metals with sulphides insoluble in dilute mineral acids. It is then boiled with a few drops of hydrogen peroxide solution, rendered alkaline with ammonia, and again treated with hydrogen peroxide to carry down manganese with the hydroxides of the iron group. The precipitate is filtered off, redissolved, and reprecipitated to recover any co-precipitated zinc. The filtrates are united and an excess of potassium hydroxide added—that is to say, at least as much as is equivalent to the zinc present. Excess of benzene-sulphonic acid is next added, the solution heated to boiling, and a current of hydrogen sulphide led through it until it is quite cold. The application of the method to the separation of zinc from nickel and cobalt, calcium and magnesium will form the subject of another paper. G. C. J.

APPARATUS, ETC.

Apparatus for Estimating Dust in Gas. C. T. Nesbitt. (*Chem. News*, 1912, **106**, 235.)—Two smooth, unribbed funnels, the edges of which fit closely, are clamped together in an apparatus consisting essentially of two wooden rings joined over the funnels by means of (1) a strip metal hinge, and (2) an ordinary screw-clip, by which the connection can be made gas-tight. Between the edges of the funnels a circular ring of thin rubber (about $\frac{3}{16}$ inch wide) is placed, to prevent leakage. This rubber is best glued to the back funnel. A piece of copper gauze is bent and placed in the back funnel so that its flat surface is flush with the edges of the funnel, and a filter-paper is placed on the rubber ring and gauze, when the other funnel is fixed in position and the whole clamped together. The first funnel is tared before use, about 100 litres of gas run through the apparatus, and, after drying, the funnel is weighed again. The filter-paper with the dust on it is burnt, and the weight of dust so found, added to the increase of weight of the funnel, gives the total dust in the quantity of gas used. A satisfactory filter-paper is C. S. and S. No. 589 "Blue Band." A. R. T.



Simple Extraction Apparatus. L. Kalusky. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1912, **24**, 623-624.)—A small perforated cup provided with three legs (see figure) is placed in the extraction flask by means of the rod; this cup supports the thimble containing the substance to be extracted, and the flask is attached directly to the condenser by means of a cork. The upper end of the thimble rests against the neck of the flask. The quantity of solvent added to the flask should be such that it does not touch the bottom of the thimble. As the extraction is made at the boiling-point of the solvent, it is claimed that the rate of extraction of a constituent from a substance is more rapid than in the ordinary extraction methods. W. P. S.

Prevention of Emulsification in Extractions by Immiscible Solvents. G. H. Meeker. (*Eighth Inter. Cong. App. Chem.*, 1912, vol. 17, 45-49.)—The apparatus described consists of a cylindrical separator into which dips a vertical shaft provided near its lower end with two sets of propellers, one of which is adjusted so that it is completely immersed in the lower liquid, whilst the other revolves in the upper liquid. The propeller blades are of curvilinear outline, and, when the shaft is driven at a suitable speed by means of a cord connected with a motor, cause the upper liquid to be driven downwards, and the lower liquid upwards, so that fresh surfaces are continually brought into contact without sufficient interagitation being caused to produce an emulsion. The extraction of alkaloids by means of this apparatus has been found to be more complete than by the older "shaking-out" method.

C. A. M.

Determination of the Permeability of Soils to Water. J. W. Leather. (*Eighth Int. Cong. App. Chem.*, 1912, vol. 15, 155-158.)—The rate at which water can flow through a short column of soil when a constant supply is maintained on the surface varies considerably with different soils. Some of the alkali lands in India are highly impervious. To enable comparisons to be made with ordinary soils, the author has designed a special and somewhat elaborate machine (two illustrations are given in the paper) to fill soils uniformly into metal cylinders measuring 3 inches high by 2 inches diameter, and provided with a false bottom of brass wire gauze. Water is then poured on the surface, and after it has once commenced to flow from the lower surface the rate of flow is measured, the hydrostatic "head" being maintained within desired limits.

H. F. E. H.

Unusual Case of Specific Gravity. A. L. Hyde. (*J. Amer. Chem. Soc.*, 1912, **34**, 1507-1509.)—The author has noticed that when paranitrotoluene (sp. gr. 1·2856) is dissolved in carbon disulphide (sp. gr. 1·2660 at 20° C.) the resulting solution is lighter in gravity than pure carbon disulphide. This is contrary to the usual rule that, when a solid heavier than a liquid is dissolved in the liquid, the resulting solution has a higher specific gravity than the pure liquid. Thus, it was found that a solution of paranitrotoluene in carbon disulphide of 20·9 per cent. strength had a sp. gr. of 1·2370 at 20° C., and one of 53·0 per cent. a sp. gr. of 1·1988, and the decrease of sp. gr. is nearly proportional to the percentage of solid in solution. Further experiments have shown that the tendency of both carbon disulphide and of paranitrotoluene is to increase the volume of solutions in which they are constituents, and where both are present an actual lowering of sp. gr. results.

A. R. T.

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REVIEWS.

Fatty Foods. By E. Richards Bolton and Cecil Revis. London: J. and A. Churchill. 1913. Pp. xii + 371. Price 10s. 6d.

The plan of this book presents several original features, which show considerable boldness on the part of the authors, who appear conscious that their departure from the usual methods invites criticism, and who are somewhat aggressively apologetic.

The book certainly invites criticism, but it must be on the whole a favourable criticism. The novel features are—first, the wholesale rejection of time-honoured methods of examination; secondly, the ignoring of the abnormal results occasionally yielded by rare specimens; thirdly, the employment of the method of ratios between the various constants for diagnostic purposes; and, lastly, the bold advocacy of scientific guesswork founded upon such data as commercial values.

As to the first feature, the authors appear to have been wise in their choice of methods. It goes without saying that it is worse than useless to employ a method which only gives information which either is less valuable or is obtained with greater labour than that yielded by other methods, and though opinions may possibly differ as to whether the very best methods have been chosen, the principle is thoroughly sound.

The second feature will probably render the opinion formed by following the advice given in this work more thoroughly sound than if all the extreme variations were taken into account. This may seem paradoxical, but it is better to form a correct judgment in the majority of instances, and blunder on the very rare occasion, than to be constantly doubtful. The one objection, which the public analyst will perhaps appreciate more than his technical colleague, is that the extreme values are likely to be insisted upon in cross-examination.

Regarding the third, nothing but praise must be given to the method of employing the ratios between the various figures determined for diagnostic purposes, and the charts given should materially assist the solution of the problems for which they are designed.

As to the scientific guesswork, this is, of course, employed by every expert, but it is refreshing to see such a point made of it in a textbook. The numerous examples, if carefully studied, should prove of great value in training the mind of the chemist who has not hitherto fully grasped this deductive process. They, as well as the descriptions of the methods, are models of lucidity, and the whole book, despite occasional literary lapses, is written in language which is a pleasure to read.

The illustrations are fairly good, but do not always give quite a correct idea. For instance, the drawing of the earth-nut appears to show that the pod has black markings on it, which is, of course, not the case; and the photomicrographs, owing to their small size and to their reproduction in half-tone, have lost a great deal of their detail.

The book is well got up, well bound, and singularly free from typographical errors, and is interleaved at intervals with blank pages for the user's own notes; these points, small though they are, add to the value of a most useful laboratory companion.

H. Droop Richmond.

Eaux Douces et Eaux Minérales. By F. Diénert. Paris: Ch. Béranger. 1912. Pp. 354 + Table of Contents. Price 6s.

This volume is one of the series of "Manuels Pratiques d'Analyses Chimiques," published under the direction of MM. Bordas and Roux. Of these nine or ten have already appeared, and the complete series will comprise twenty-four volumes. The series is intended more especially for the use of analysts and experts attached to official laboratories in France, and the volume under review is written by the chief of the "Service de la Surveillance des Eaux d'Alimentation de Paris." About half of the book is devoted to the methods of analysis, chemical, physical, and bacteriological, of "soft" and mineral waters, and tables are given showing the composition of almost all the better-known Continental mineral waters. The analytical methods given are mainly confined to those which have received "official" sanction in France; and in several sections, notably in that dealing with the estimation of nitrates, no mention is made of several well-known methods which are commonly employed by water analysts. Such analytical methods as are given are, for the most part, clearly described, and the book would appear to be admirably suited to the purpose for which it is intended; but from the point of view of the water analyst, who is called upon to express an opinion as to the quality of a water-supply, and to suggest the most efficient means of purification, it is to be regretted that no actual figures are given of the results of analysis of waters of different types, which would illustrate the principles upon which such an opinion should be based. More detailed criticism of and comparisons between the different analytical methods employed, and references to recent work on water purification, such as is to be found in the annual reports of the Metropolitan Water Board, would have materially enhanced the value of what is otherwise a useful book. The chapters on investigation in the "field," sampling, etc., contain some interesting and suggestive details, many of which are apt to be overlooked when collecting samples for analysis. The volume concludes with an appendix, containing information as to the more important legislation enacted in respect of the sale of mineral waters in France. G. W. Monier-Williams.

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INSTITUTE OF CHEMISTRY OF GREAT BRITAIN AND IRELAND.

Final Examination for the Associateship in the Branch of Biological Chemistry. October 14 to 18, 1912.

The following candidates passed: Robert Philip Keith, Eric Richards, and Frederic Robinson.

FEBRUARY, 1913. Vol. XXXVIII., No. 443

THE ANALYST.

OBITUARY.

A. C. CLAUDET.

THE sudden and unexpected death of Mr. A. C. Claudet from pneumonia, on January 18, came as a great shock to mining and metallurgical men amongst whom he was so well known. For some time he had been suffering with a bad cold, but nothing serious was anticipated until about a week before his death, when the cold developed into pneumonia. Mr. Claudet was born on June 9, 1855, and was the eldest son of the late Mr. Frederick Claudet of London and Cannes, the founder of the well-known firm of assayers and metallurgists. He received his technical education at the Royal School of Mines, where he took his Associateship in 1878. He was an original member of the Institution of Mining and Metallurgy, treasurer since the date of its foundation in 1892, president in 1906-1907, and had been one of the trustees for a number of years. He served on the Council of the Institute of Chemistry and the Faraday Society, and on several other societies, and identified himself in every way with scientific progress. He stood in the front rank of British metallurgists, and was one of the official assayers to the Bank of England.

For many years he had taken an active part in the advancement of technical education, and was especially interested in the reorganisation of the Royal School of Mines, and in the rebuilding and equipment of the new laboratories in connection with the Imperial College of Science and Technology. He was one of the most influential members of the Executive Committee of the Bessemer Memorial Fund, and it was largely due to his influence that this fund was applied to the equipment of the Bessemer Laboratory.

He took the keenest interest in young students, and, in conjunction with Mr. Henning Jennings of Washington, established "A Post Graduates' Grant Fund," under the auspices of the Institution of Mining and Metallurgy, to supplement the scholarships given by that institution to assist graduates to obtain practical experience in mines and works in different parts of the world; many young men owe their start in their professional career to the assistance thus afforded by his kindly forethought.

He also identified himself with the Students' Union at the Imperial College of Science and Technology, both by giving it his financial support and by personally persuading many of his friends to help, and students of the Royal School of Mines have lost one of their best friends.

To those who were brought into touch with him professionally, and still more those who were privileged to number him among their personal friends, his loss seems irreparable. He was ever one of the kindliest of men, always looking out to lend a helping hand in a quiet, unobtrusive way, either by personal service or financially in cases of need. Even those who were intimately associated with him never knew how many and varied were his activities in the interests of others. He will be sadly missed.

F. W. HARBORD.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

DELICATE COLOUR REACTIONS OF GOLD SALTS.

BY J. E. SAUL, F.I.C.

IT is found that dilute solutions of certain aromatic reducing agents added to solutions of gold salts result in the formation of coloured colloidal solutions of gold. Quinol, pyrogallol, gallotannic acid, *p*-oxyphenylaminoacetic acid (glycin), and salts of phenylhydrazine, 1 : 2 : 4 diaminophenol (amidol), the monomethyl derivatives of *m*- and *p*- (metol), and *o*-aminophenol, associated with quinol in (ortol), and *m*-phenylenediamine, may be mentioned amongst others as producing these reactions.

If one volume of a 0·1 per cent. solution of any of the above compounds is added to ten volumes of a solution containing 0·002 per cent. of auric chloride, a violet-coloured colloidal solution of gold is produced in the course of a couple of minutes. The colours vary according to the reagents used and the strength of the solutions. In the case of gallotannic acid the colloidal solution in that dilution is rose-coloured, and the tint takes a longer time to develop. The colours are generally very beautiful and effective, in some cases much more than in others. The colours yielded with (metol) and quinol, for example, are particularly fine.

In studying these reactions, the writer has observed that *p*-phenylenediamine acts abnormally, and, as the resulting reaction is of extraordinary delicacy, he ventures to suggest its use as a test for traces of gold salts. In this case a dark yellowish-green solution results, which, after the lapse of some time, undergoes further changes. In using the test it is suggested that a weaker solution of the gold salt be employed. One volume of a 0·1 per cent. solution of *p*-phenylenediamine hydrochloride should be added to ten volumes of a solution containing 0·0005 per cent. of the gold chloride. A distinct greenish-yellow coloration results, and this is still quite visible in a solution containing only 0·0001 per cent. of the gold salt. The reaction appears to transcend in delicacy any of the violet reactions before referred to, and it may be found useful occasionally in analysis.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Estimation of Boric Acid in Charcuterie. V. Villavecchia and I. Barboni. (*Ann. Lab. Chim. Cent. delle Gabelle*, 1912, **6**, 27-68.)—The author points out that various samples of Italian charcuterie thus examined were found to contain from 0·004 to 0·020 per cent. of boric acid. It was found, however, that most of the samples of Italian salt contained appreciable amounts of boric acid, reaching a maximum quantity of 3·25 per cent. in Salsomaggiore salt. It is therefore suggested that a limit of 0·02 per cent. should be fixed as the maximum permissible limit for boric acid unintentionally introduced with the salt into flesh products. C. A. M.

Detection of Citric Acid in Wines. W. Fresenius and L. Grünhut. (*Zeitsch. anal. Chem.*, 1913, **52**, 31-35.)—An investigation of various methods which have been proposed for the detection of citric acid in wine has been carried out by the authors, who found that Deniges' method (conversion of citric acid, by oxidation with permanganate, into acetonedicarboxylic acid and precipitation of the latter as its mercury compound—*cf.* L. Gowing Scopes, ANALYST, 1913, 12) is untrustworthy, positive reactions being obtained in cases where citric acid was absent. Krug's modification of Moslinger's test yields reliable results. In this the wine, after removal of tartaric acid and alcohol, is treated with acetic acid and lead acetate, heated and cooled, and a turbidity then obtained indicates the presence of citric acid. If the amount of fixed acids exceeds 0·28 per cent., the 50 c.c. of wine taken for the test should be evaporated, not to 10 c.c., but to a correspondingly larger volume before the lead acetate is added. For instance, a wine containing x grm. of fixed acids per 100 c.c. should, after the removal of the tartaric acid and alcohol, be brought to a volume of 37·5 x c.c.; this dilution prevents the interference of malic acid. The test will readily detect the presence of 0·05 grm. of citric acid per 100 c.c. of wine. Schindler's test is also trustworthy, but somewhat less sensitive than that recommended by Krug; it consists in treating 50 c.c. of the wine with ammonia, adding 3 c.c. of alcohol and a few c.c. of 10 per cent. barium chloride solution. After the lapse of twenty-four hours the precipitate is collected on a filter, then rinsed with not more than 15 c.c. of water into a beaker, and the boiling mixture is treated with sulphuric acid. The barium sulphate is separated by filtration, and to the filtrate are added 1·5 c.c. of basic lead acetate solution and 1·5 c.c. of a saturated lead acetate solution; the mixture is boiled, and filtered while hot. If the filtrate becomes turbid, the presence of citric acid is indicated. W. P. S.

Estimation of the Quality of Flour. G. Savini and G. Silvestri. (*Ann. Lab. Chim. Cent. delle Gabelle*, 1912, **6**, 425-433.)—A simple method of estimating the quality (fineness) of flour is based upon the measurement of the quantity of deposit obtained on shaking the sample with water under definite conditions. The method found most suitable was to place 5 grms. of the flour in a graduated stoppered cylinder with an internal diameter of 20 mm., and to add 25 c.c. of water containing 5 per cent. of glycerol (sp. gr. 1·25) and 0·25 per cent. of 40 per cent. formaldehyde solution. The cylinder was next shaken vigorously for exactly a minute, then allowed to stand for thirty minutes, and the volume of deposit read upon the scale. The smaller the proportion of deposit, the better the quality of the flour. For example, in the best qualities the amounts ranged from about 10·8 to 12·6 c.c., whilst inferior qualities yielded from 19 to 27·6 c.c. As a rule, the amount of the deposit showed a certain parallelism with the percentage of ash, and in the case of samples from two mills, when the third grades gave somewhat better results in this test than the second grades, the percentages of ash in the latter were also respectively lower. Test experiments were made with mixtures of a good and poor grade of flour in various proportions, with results of which the following are typical. The good flour contained 0·55 per cent. of ash on the dried substance, and yielded a deposit of 12 c.c.; whilst the inferior flour contained 2·7 per cent. of ash, and gave a deposit of 25 c.c.:

Per cent. of inferior flour	2·5	5·0	7·5	10·0	20·0	30·0	50·0
Volume of deposit in c.c.	12·2	12·6	13·0	14·2	18·4	20·6	23·2

The influence of the addition of washed bran (up to 2 per cent.) and of wheat oil to the fine flour was also very pronounced. In the case of greatly decomposed flour the deposit had not formed after the cylinder had stood for thirty minutes.

C. A. M.

Essential Oil of Jamaica Ginger. F. D. Dodge. (*Eighth Int. Cong. App. Chem.*, 1912, Vol. 6, 77-80.)—The essential oil of Jamaica ginger contains camphene, *l*-phellandrene, sesquiterpine, and a crystalline saturated aldehyde. This aldehyde, the proportion of which varies from 2 to 5 per cent., may be isolated from the first fractions obtained by distilling the oil with steam or *in vacuo*, by shaking them with a fifth of their volume of 15 per cent. sodium bisulphite solution (free from excess of sulphur dioxide), and leaving the mixture for several days at a low temperature. The precipitate is then separated with the aid of a filter-pump, washed with benzene, and dried in the air till odourless, after which it is mixed with a considerable volume of water and the calculated amount of sodium bicarbonate, and the aldehyde distilled in a current of steam. As thus obtained it is a colourless, optically inactive liquid, with the characteristic orange odour of decylic aldehyde. It has a sp. gr. of 0·828 at 15° C., and yields an oxime melting at 63° C. It is probably identical with the decylic aldehyde contained in the essential oils of orange, iris, coriander, etc.

C. A. M.

Estimation of Methyl Alcohol in Spirits. J. Hetper. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1912, **24**, 731-737.)—The total quantity of alcohol in the sample is first estimated, and a portion of the solution is then oxidised with permanganate, the amounts of the two alcohols (methyl and ethyl) being calculated from the quantity of permanganate reduced. In phosphoric acid solution methyl alcohol is oxidised by permanganate to carbon dioxide and water, whilst ethyl alcohol yields acetic acid, which is not further attacked by permanganate. One grm. of methyl alcohol requires 187·5 c.c. of $\frac{N}{1}$ permanganate for oxidation, and 1 grm. of ethyl alcohol only 87 c.c. of this solution. For the estimation of the total alcoholic strength of a spirit, advantage is taken of the fact that the specific gravities of aqueous solutions of methyl or ethyl alcohols are practically identical when the amount of the alcohols is about 50 per cent. by weight. For instance, a 50 per cent. methyl alcohol solution has a sp. gr. of 0·9185, whilst the sp. gr. of a 50 per cent. ethyl alcohol solution is 0·9184. The spirit under examination is, therefore, either diluted or concentrated by distillation until it has a sp. gr. of from 0·910 to 0·925; the actual sp. gr. is then determined, and the alcoholic strength found by reference to tables. About 2 c.c. of this alcoholic solution are then weighed accurately and diluted to 200 c.c.; 10 c.c. of the latter solution are introduced into a flask in which have been placed previously 30 c.c. of $\frac{N}{2}$ potassium permanganate solution (containing 40 grms. of crystallised phosphoric acid per litre) and 60 c.c. of water. The flask is attached to a reflux condenser and heated for two hours on a boiling water-bath. The condenser tube should be rinsed down a few times with water during this period. To the hot solution are then added 30 c.c. of $\frac{N}{2}$ oxalic acid solution, and the excess of the latter is titrated with $\frac{N}{2}$ permanganate solution. The amount of the methyl alcohol is found from the amount of permanganate consumed, and, the total alcoholic strength being known, the amount of the two alcohols in the original spirit may be calculated. The process cannot be applied to spirits containing large quantities of ethyl acetate or furfural. W. P. S.

Rapid Control Method for the Determination of Oil in Grains. E. P. Harding and L. L. Nye. (*J. Ind. Eng. Chem.*, 1912, 4, 895.)—The considerable length of time required for the extraction of fats by means of ether or light petroleum, and the necessity for making the extraction on a moisture-free sample, led to the substitution of carbon tetrachloride as a solvent. This substance has been used by Bryant, but the difficulty of removing the last traces of solvent is a disadvantage. In the author's method, 1 to 3 grms. of the sample are heated with 25 c.c. of carbon tetrachloride in a thick-walled cylinder—$1\frac{1}{4}$ by $7\frac{1}{2}$ inches —under a reflux condenser, 12·5 c.c. of carbon tetrachloride distilled off, the cylinder cooled, and 25 c.c. of absolute alcohol, 12·5 c.c. of water, and 50 c.c. of light petroleum, added, the cylinder being shaken after each addition. It is then rotated in a centrifugal machine for ten minutes at 1,000 revolutions per minute. The light petroleum layer is then blown off through a filter into a tared flask, and the solvent distilled off. The extraction of the contents of the cylinder is then repeated twice, but with 5 c.c. of carbon tetrachloride, 5 c.c. of absolute alcohol, and 50 c.c. of light petroleum; the filter is washed into the flask, the solvent distilled off, and the flask heated for successive periods of fifteen minutes in boiling water in a Soxhlet until its weight is constant. The results compare favourably with those obtained with ether or light petroleum, and the oil extracted is purer, as its higher iodine value shows. A complete determination can be carried out in two and a half hours. O. E. M.

Detection of Earthnut (Arachis) Oil in Olive Oil by the Franz-Adler Method. H. Lüers. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1912, **24**, 683-684.)—Two samples of olive oil examined by the author yielded a precipitate at 16° C. when submitted to the test proposed by Franz and Adler for the detection of earthnut oil (*cf.* ANALYST, 1912, **37**, 487); this oil was, however, proved to be absent by means of the lead-soap method described by Tortelli and Ruggeri (ANALYST, 1898, **23**, 273). The oils were then submitted to further investigation, and it was found that they contained unusually high proportions of myristin. The precipitate obtained in the Franz-Adler test consisted of the acid potassium salt of myristic acid ($KC_{14}H_{27}O_2.C_{14}H_{28}O_2$). The formation of this salt in the course of the test may be prevented by the addition of 3 drops of glacial acetic acid (in addition to the prescribed quantity of dilute acetic acid) to the saponified oil. If it be preferred, the test may be carried out in the usual manner, and the additional amount of acetic acid used only in those cases where a preliminary test gives a turbidity indicating the presence of earthnut oil. W. P. S.

Adulterated Peppercorns. A. Troccoli and G. Verona-Rinati. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1912, **24**, 736-741.)—Certain samples of peppercorns examined by the author were found to contain seeds of *Rhamnus catharticus* (buckthorn) and *Schinus molle* (so-called American, or false, pepper). In one instance the sample consisted wholly of the latter seeds. Other samples consisted of small peppercorns coated with a mixture consisting of pepper offal, talc, chalk, charcoal, starch, and dextrin. The weight of this coating amounted to from 25 to 70 per cent. of the weight of the whole corns. W. P. S.

Reaction of Peru Balsam. C. Griebal. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1912, **24**, 688.)—When a few mgrms. of Peru balsam are treated with 2 or 3 drops of alcoholic potassium hydroxide solution, a characteristic odour resembling that of ethyl benzoate is produced. This is due to the saponification of the cinnamein, which consists of a mixture of benzyl benzoate and benzyl cinnamate. For the detection of Peru balsam in preparations consisting principally of a fatty basis, the cinnamein may be separated by subjecting the preparation to steam-distillation, extracting the distillate with ether, evaporating the ethereal solution, and testing the residue as described. Cinnamein is scarcely volatile at 100° C., and essential oils and certain other substances which might interfere with the test may be removed by heating the cinnamein on a water-bath. Besides Peru balsam, a few other substances of vegetable origin—notably, Tolu balsam and benzoin—contain esters of benzoic acid, and yield an odour when treated with alkali. These two drugs contain, however, but a small proportion of esters and large quantities of free acids (cinnamic and benzoic acids) and resin. W. P. S.

Estimation of Phosphorus in Lecithin. P. Freundler. (*Bull. Soc. Chim.*, 1912, **11**, 1041-1043.)—It is recommended that the substance be first treated with nitric acid, and the oxidation then completed by the addition of potassium permanganate. About 2 grms. of the lecithin are heated on a water-bath with 50 c.c. of fuming nitric acid for two to three hours, or until all action ceases and a pale yellow solution is obtained. After cooling, potassium permanganate is added in small quantities at a time until the permanganate colour in the liquid is no longer discharged; about 30 grms. of the salt will be required. The mixture is then diluted with water to 150 c.c., the precipitated manganese dioxide is dissolved by the addition of sodium nitrite, and the solution is evaporated to a syrupy consistence. This residue is dissolved in about 70 c.c. of water, and the phosphoric acid is precipitated with molybdic acid solution in the usual way. W. P. S.

Quinine Assay. P. J. Kruysse. (*Chemist and Druggist*, 1913, **82**, 24.)—The method utilises the insolubility of quinine nitroprusside in order to separate it from other cinchona alkaloids. Five grms. of cinchona bark in fine powder are made into a paste with 3·5 c.c. of water and 1 c.c. of 10 per cent. ammonia. The mixture, after the addition of 2·5 grms. of slaked lime, is extracted for two hours in a Soxhlet with acetone. The alkaloids in the residue, after drying on the water-bath, are dissolved in 25 c.c. of 1 or 1·5 per cent. hot hydrochloric acid, and the solution is filtered with successive small washings into a separator, where it is made alkaline with caustic potash, and shaken out with two successive quantities (about 200 c.c.) of ether. The alkaloids are removed from the ether by agitation with small quantities of 1·5 per cent. hydrochloric acid, and subsequently neutralised with caustic potash. The liquid, which should now measure about 50 c.c., is heated to boiling, and 0·5 grm. of ammonium oxalate is added, the resulting crystals being filtered and washed, and finally dissolved on the filter with 5 c.c. of 2 per cent. hydrochloric acid added drop by drop. Washing of the filter paper is continued till about 50 c.c. have been collected, and $\frac{N}{2}$ ammonia is added in quantity just insufficient to cause precipitation. A little calcium chloride is now added, and the precipitate of calcium oxalate is filtered off and washed with water. The filtrate is next exactly neutralised with ammonia, heated, and 0·5 grm. of sodium nitroprusside added to it, the whole then being allowed to cool. The crystals obtained are filtered, and dried on the paper at 100° C. The weight so obtained is multiplied by 1·03, and to this is added 0·05 grm. (the equivalent of 20 mgrms. of quinine sulphate lost as oxalate, and 30 mgrms. as nitroprusside). The result is the weight of quinine calculated as sulphate which can be obtained from 5 grms. of the bark. H. F. E. H.

Estimation of Traces of Iron in Cane and Beet Sugar Factory and Refinery Products. J. J. Eastick, J. P. Ogilvie, and J. H. Lindfield. (*Int. Sugar J.*, 1912, **14**, 428-435.)—The method recommended is that known as the sulphide colorimetric process, and has been used by Winkler in other branches of analysis (*cf.* ANALYST, 1903, **28**, 16). It has the advantage over the thiocyanate and ferrocyanide colorimetric methods in that it can be applied directly to certain sugar products without the inconvenience of previous incineration of the sample. The estimation is carried out as follows: A standard iron solution is prepared by dissolving 10 grms. of ferrous sulphate in water, adding a few drops of sulphuric acid, and diluting the mixture to 1 litre. If 2 c.c. of this solution be diluted to 500 c.c., a solution is obtained each c.c. of which will contain 0·00002 grm. of iron

(Fe). In the case of light-coloured sugar products, from 3 to 10 grms. are dissolved in 100 c.c. of water in a graduated 100 c.c. cylinder, 2 c.c. of freshly-prepared ammonium sulphide are added, and the coloration obtained is compared with that produced, under similar conditions, by a known quantity of the dilute ferrous sulphate solution. Dark-coloured products must, of course, be incinerated before the test is applied; sulphuric acid is added previous to the ignition, and the ash is dissolved in the least possible quantity of hydrochloric acid, the solution being then diluted with water to 100 c.c. Results of experiments are given, which show that the same amount of iron is obtained whether it be estimated directly in a light-coloured sugar product or in the ash of the same. As regards the influence of the presence of other metals—notably lead and copper—on the estimation, it was found that lead yields a light brown coloration which is characteristic, whilst copper gives no appreciable coloration. The presence of these metals is, however, quite exceptional, and it is a simple matter to detect them by a preliminary qualitative examination.

It is pointed out that if, instead of water alone, a 50 per cent. sucrose solution is employed in the preparation of the ferrous sulphate solution, the latter will keep almost indefinitely. W. P. S.

Roselle. D. S. Pratt. (*Philippine J. Science*, 1912, **7**, 201-205.—The roselle plant (*Hibiscus sabdariffa*, L.) has recently been introduced into the Philippine Islands, and is grown chiefly for its edible calyx, which, after the plant has flowered, enlarges considerably, and is used in making jam or jelly of a brilliant red colour and pleasant acid taste. The following are analyses of two samples of roselle and one of cranberry:

	ROSELLE.		CRANBERRY.
	U.S.A.	Philippines.	
	Per Cent.	Per Cent.	Per Cent.
Water	88·91	82·49	88·53
Solids	11·09	17·51	11·47
Ash	0·89	1·26	0·25
Marc (insol. matter)	6·67	7·39	4·60
Malic acid	2·77	3·31	2·74
Invert sugar	0·33	0·82	1·90
Sucrose	0·03	0·24	0·10
Benzoic acid	Absent	Absent	Present

The Philippine roselle (both calyx and leaves, etc.) contains the dextro-rotatory form of malic acid, not hitherto found in nature, though the lævo-rotatory form is commonly present in fruits. The entire plant may be used for preparing a flavouring syrup and a wine. A. R. T.

Detection and Estimation of Xanthine Bases in Cocoa, Tea, Coffee, and their Preparations. S. Camilla and C. Pertusi. (*Ann. Lab. Chim. Cent. delle Gabelle*, 1912, **6**, 611-626.)—A sensitive test for the presence of xanthic bases (caffeine, theobromine, etc.) is based upon the fact that they are readily oxidised to carbylamine, which may be recognised by its characteristic odour. A small quantity of the substance under examination is mixed with a few drops of a concentrated solution of potassium hydroxide, followed by a few drops of a saturated solution of potassium permanganate, and the mixture gently heated. By means of this test it was found that ordinary commercial cocoa butter contains traces of xanthine bases. *Estimation of Xanthine Bases.*—Ten grms. of, *e.g.*, cocoa or chocolate are boiled for thirty minutes with 150 c.c. of water and 50 c.c. of $\frac{N}{1}$ sulphuric acid, and the liquid then diluted to 500 c.c., heated on the water-bath, and filtered while hot. The filtrate is cooled, and 250 c.c. of it neutralised with magnesium oxide and evaporated to about 80 c.c., after which it is transferred to a special form of Soxhlet's extractor, and extracted for two to three hours with boiling carbon tetrachloride. Or it may be shaken in a separating funnel with three times its volume of carbon tetrachloride. This solvent extracts the caffeine and a little of the theobromine simultaneously with the fat, whilst the bulk of the theobromine remains in the aqueous layer. The carbon tetrachloride is evaporated after the addition of a little paraffin wax, and the residue repeatedly treated with small quantities (25 to 30 c.c. at a time) of boiling water slightly acidified with sulphuric acid. The aqueous extracts are filtered and added to the original aqueous layer, and the whole mixed with 5 grms. of magnesium oxide and evaporated to dryness. The residual mass is then powdered and extracted four or five times with successive portions (100 c.c.) of boiling chloroform, for ten minutes each time. If the extracted theobromine and caffeine are not perfectly white and well crystallised, they are purified by treating their aqueous solution with 1 per cent. of cold potassium permanganate solution, and again extracting them with chloroform in a separating funnel. C. A. M.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Action of "Activated" Aluminium on Alkaloid Extracts and its Employment in Toxicology. E. Kohn-Abrest. (*Comptes rend.*, 1912, **155**, 1179-1181.)—In many cases "activated" aluminium may be substituted with advantage for basic lead acetate or animal charcoal for the clarification of organic liquids. Thin sheets of aluminium foil are immersed for three minutes in a 1 per cent. solution of mercuric chloride. In presence of water this "activated" foil causes a gentle evolution of hydrogen, the decolorising and preservative action of which is supplemented by the absorptive affinity of the aluminium oxide produced. Such foil has been used with much success in the treatment of the aqueous-alcoholic extracts of putrid or putrifiable animal organs for the toxicological separation of alkaloids. A series of experiments was first performed with solutions of the pure alkaloids in a mixture of 80 c.c. of alcohol, 20 c.c. of water, and 1 grm. of tartaric acid, 0·1 grm. of alkaloid being added in each case. These solutions were each treated for twenty-four hours with a piece of foil weighing 3 to 4 grms.; the alkaloids were then estimated by approved methods. In the case of morphine, heroine, narcotine, codeine, atropine, brucine, narceine, and antipyrine, the quantity of alkaloid recovered was 90 to 100 per cent.; in the case of caffeine and veratrine, 80 to 85 per cent.; and with strychnine, quinine, and cocaine, only 50 to 60 per cent. Nicotine, on the other hand, was almost completely absorbed by the aluminium hydroxide. Further experiments were made by extracting animal organs with alcohol acidified with tartaric acid in the usual manner. The aqueous-alcoholic extracts obtained were treated for twenty-four hours with 10 to 12 grms. of the active foil per litre of liquid. The liquid should remain acid during the whole period, and when filtered on the pump should have a straw-yellow colour; if not, the treatment should be repeated with fresh foil. Tests made with morphine and strychnine in animal viscera yielded in all cases perfectly satisfactory results, even when the quantity of alkaloid added was as low as 0·0015 grm. per 100 grms. The extracts treated in the above manner are always exceptionally pure, and the characteristic reactions of the alkaloids are obtained without the interference of disturbing impurities, so that, although in certain cases some loss of alkaloid is unavoidable, it is nevertheless compensated by the increased sharpness of the tests. J. F. B.

Estimation and Detection of Small Quantities of Methyl Alcohol in Blood and Animal Tissues. M. Nicloux. (*Eighth Int. Cong. App. Chem.*, 1912, vol. 19, 259-260.)—The blood or finely-divided tissue is mixed from six to eight times its weight of a saturated picric acid solution, and distilled until one-fifth of the volume of the mixture has been collected as distillate. This distillate is then oxidised with a potassium bichromate solution containing 19 grms. of the salt per litre; the carbon dioxide resulting from the oxidation is collected and weighed, and the quantity of bichromate used is ascertained in the usual manner. If the ratio $O_2 : CO_2$ is approximately equal to 0·915, methyl alcohol is present in the blood or tissue, and the quantity of the alcohol may be calculated from the amount of bichromate required for the oxidation. W. P. S.

Investigations on the Quantitative Reduction of Methylene Blue by Milk Bacteria, and the Use of this Dye for Determining the Condition of Milk. E. B. Tred. (*Zentralbl. f. Bakter. u. Parasitenk.* 1912, **35**, II. 391; through *Chem. Zentralbl.*, 1912, II., 1991.)—The flora of milk is specially rich in micro-organisms capable of reducing methylene blue. Of twenty-two species, twenty-one are able to effect this reduction, which takes place more rapidly in milk than in bouillon; the velocity is a linear function of the temperature, increasing with rise of temperature up to 37° C. The reduction in a culture inoculated into a new medium is directly proportional to the growth of the bacteria, and ceases with the exhaustion of the medium. Each species of bacteria has a different coefficient of reduction of methylene blue. Multiplication of organisms and reduction of dyestuff give, for all species, curves of the same general form; their courses are therefore parallel. Peroxydase exists ready formed in the milk; it is not a product of the growth of bacteria, as are catalase and reductase. The reduction of Schardinger's reagent is due to aldehydecatalase, the enzyme which promotes the reduction of methylene blue by formaldehyde (Smidt, *Hygien. Rundsch.*, 1905, **14**, 1138). The reduction of methylene blue in the absence of formaldehyde is complicated; it is probable that intracellular and extracellular products take part in it. The reduction test affords an easy method for the approximate determination of the number of bacteria in milk. Thus, if reduction is effected within fifteen minutes, the milk contains 15 to 50 million per c.c.; if seven or more hours are required, less than 1 million.

O. E. M.

Estimation of Glycogen in Liver. H. Bierry and Z. Gruzewska. (*Comptes rend.*, 1912, **155**, 1559-1561.)—Twenty-five grms. of the crushed liver are mixed with 25 c.c. of 35 per cent. potassium hydroxide solution, and the flask heated on the water-bath until the tissue has completely dissolved, after which the mixture is heated in an autoclave at 120° C. for thirty minutes. The liquid is then cooled, neutralised with hydrochloric acid, with turmeric as indicator, and, after the addition of 5 c.c. of hydrochloric acid of sp. gr. 1·18, made up to 100 c.c., and again heated for thirty minutes at 120° C. It is now cooled, transferred to a beaker, neutralised with sodium hydroxide, and treated, little by little, with mercuric nitrate, with continual stirring, to precipitate the proteins. Finally, it is made up to 300 c.c. and filtered, the filtrate treated with zinc powder, and allowed to stand for several hours until colourless, and the dextrose then estimated by Bertrand's method in 5, 10, or 20 c.c. of the liquid. The weight of dextrose multiplied by the factor 0·927 gives the weight of glycogen. The results thus obtained are somewhat higher than those given by Pflüger's method. In the new method the nucleoproteins are partially destroyed, and the amount of *l*-xylose that they yield in the hydrolysis is less than 0·006 grm. from 25 grms. of liver.

C. A. M.

Estimation of Mercury in Textile Materials. L. L. Lloyd and W. M. Gardner. (*J. Soc. Chem. Ind.*, 1912, **31**, 1109-1112.)—A recent case of mercurial poisoning in a hat factory led to an examination of the furs used in the manufacture. Six samples taken at random were found to contain from 0·09 to 0·175 per cent. of mercury, which had been introduced in the usual practice of the dealers of treating the furs with an acid solution of mercuric nitrate to raise the scales of the fibres and increase their felting property. In the course of the processes of manufacture some of this mercury had volatilised and caused the poisoning.

Detection of Mercury.—In fur and textile fabrics, a good method is to heat about 5 grms. of the divided material on the water-bath, with a small quantity of *aqua regia* diluted with three times its volume of water, in a 1,500 c.c. flask which is fitted with tubes for a distillation in a current of superheated steam. The acid solution in the flask is gradually treated with zinc dust, and then distilled in a current of steam heated to 160° to 170° C. The volatilised mercury is caught by two coils of bright copper gauze, one of which is placed in the upper, and the other in the lower end of the condenser. The coils are subsequently washed with alcohol, dried in the air, and gently heated in a tube, the open end of which is drawn out to form a thin constricted portion within which the mercury condenses. A minute crystal of iodine is next introduced, and the tube again gently heated. The iodine vapour coming in contact with the mercury deposit gives an orange or red coloration. Under the same conditions arsenic turns yellow, and antimony brown or yellow. If the portion of the tube containing the deposit is then cut off and heated with a drop of nitric acid in a test-tube, the iodides of arsenic and antimony will be rapidly decomposed, whilst mercuric iodide is only slowly dissolved.

Estimation of Mercury.—A weighed portion of the cloth is extracted with six successive portions of dilute *aqua regia* (1 : 2) at about 50° C., preferably in a flask fitted with an upright tube to prevent any material being carried away by the escaping gases. The residue is washed thoroughly with hot water, and the united extracts and washings made slightly alkaline with sodium hydroxide. The liquid is next saturated with hydrogen sulphide, and, after standing for sometime in a warm place, is rendered slightly acid with acetic acid, and again allowed to stand. The precipitate is then collected on a filter of special construction (*infra*), washed until free from chlorides, and then treated first with warm dilute nitric acid, and finally with boiling dilute nitric acid (1 : 3), which leaves the mercuric sulphide undissolved on the filter. Finally, the mercury is dissolved in warm dilute *aqua regia*, and it is estimated colorimetrically by comparison with a standard solution of mercury (0·1 grm. per litre). The tests are made in Nessler's tubes with a reagent containing 0·2 grm. of potassium iodide, 6·0 grms. of sodium hydroxide, and 2·0 grms. of ammonium chloride. The special filter employed consists of a glass tube terminating in a narrow constricted portion. Within the tube are arranged in layers from below:—(1) A filter-plate; (2) rough quartz; (3) glass wool; (4) a filter-plate; and (5) a layer of fine quartz, kaolin, and silicic acid. The kaolin is made into a thick cream with sodium silicate solution, and deposited on the fine quartz. The addition of a little dilute acid then liberates the silicic acid, and binds the kaolin into a gelatinous layer. This method is applicable to the estimation of mercury used as an antiseptic in sizes and dressings for textile materials. One sample of cotton-warp was found to contain 0·00015 per cent. of mercury.

C. A. M.

Detection of Pentoses in Diabetic Urine. A. Jolles. (*Zeitsch. anal. Chem.*, 1913, **52**, 104-108.)—The test proposed consists in treating the urine with phenylhydrazine hydrochloride and sodium acetate, separating the resulting precipitate and distilling it with hydrochloric acid; the distillate yields a green coloration with orcinol should pentoses be present in the urine. A negative reaction is obtained with the osazones of all the other sugars which are likely to be found in urine—namely, dextrose, lævulose, lactose, galactose, maltose, and sucrose. The test is carried out as follows: In the case of urine containing up to 5 per cent. of dextrose, 100 c.c. of the sample are heated on a water-bath for one hour with 4 grms. of phenylhydrazine hydrochloride and 8 grms. of sodium acetate. After cooling, the precipitate is separated, boiled for five minutes with 15 c.c. of water, and the hot solution is filtered. The filtrate is acidified with 6 c.c. of hydrochloric acid (sp. gr., 1·19) and distilled, 6 c.c. of distillate being collected in a test-tube. Three c.c. of this distillate are mixed with 5 c.c. of Bial's reagent (1 grm. of orcinol dissolved in 500 c.c. of 30 per cent. hydrochloric acid, with the addition of 30 drops of ferric chloride solution), and boiled for a short time; a distinct green coloration is obtained when the urine contains as little as 0·05 per cent. of pentosans. With urines containing from 5 to 10 per cent. of dextrose, double the above-mentioned quantities of phenylhydrazine hydrochloride and sodium acetate must be used. For the detection of pentoses in urine which is free from sugar, it is advisable to add about 1 per cent. of dextrose before proceeding with the test. W. P. S.

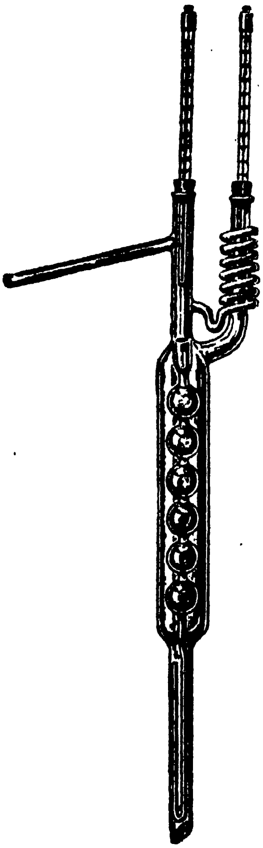
Estimation of Sugar (Dextrose) in Urine. K. K. Jarvinen. (*Zeitsch. anal. Chem.*, 1913, **52**, 14-15.)—The colorimetric method described depends on the production of a black colour when dextrose is heated with Almén-Nylander's reagent (2 grms. of bismuth subnitrate and 4 grms. of Rochelle salt dissolved in 100 grms. of 8 per cent. sodium hydroxide solution). Ten c.c. of the urine (diluted with twice its volume of water if much sugar is present) are placed in a test-tube, and 0·5 c.c. of the reagent is added; other test-tubes are prepared, each containing 10 c.c. of diluted sugar-free urine and 0·5 c.c. of the reagent, and to these tubes are added quantities (0·1 c.c., 0·2 c.c., 0·5 c.c., 1 c.c., etc., respectively) of a 1 per cent. dextrose solution. The contents of the tubes are then diluted to a volume of 12·5 c.c., and they are heated in a boiling water-bath. The coloration given by the sample of urine is then compared with that obtained in the other tubes. W. P. S.

Estimation of Uric Acid in Urine. F. Herles. (*Eighth Int. Cong. App. Chem.*, 1912, vol. 19, 141-144.)—The method proposed consists in precipitating the uric acid as ammonium urate, and titrating the latter with a mineral acid. One hundred c.c. of the urine are treated in a beaker with 36 grms. of powdered ammonium chloride, the mixture being stirred until the salt has dissolved. After the lapse of thirty minutes the precipitate is collected on a filter and washed three times with saturated ammonium chloride; the filter and precipitate are then placed in the beaker, heated to boiling with about 50 c.c. of water, and the solution is titrated with $\frac{N}{10}$ sulphuric acid, 2 drops of methyl-orange solution being added as indicator. It is advisable to add a slight excess of the acid, and to titrate this excess with $\frac{N}{10}$ alkali solution. One c.c. of $\frac{N}{10}$ acid corresponds with 0·0168 grm. of uric acid. If the urine under examination is not clear, 100 c.c. of it are warmed and treated with sodium hydroxide solution until alkaline to phenolphthaleïn. The alkaline solution is then partially evaporated, transferred to a 100 c.c. flask, cooled, diluted to 100 c.c., and filtered. Fifty c.c. of the filtrate are used for the estimation of the uric acid. Results of experiments with pure uric acid show that the method is accurate. W. P. S.

ORGANIC ANALYSIS.

Analysis of Commercial Calcium Citrate. I. Barboni. (*Ann. Lab. Chim. Cent. delle Gabelle*, 1912, **6**, 311-341.)—From the results of a critical study of the various methods proposed for the examination of commercial calcium citrate, the conclusion is drawn that the method of Spica (Analyst, 1910, **35**, 519) is the most accurate and rapid. The method of Patané, which is similar to that of Gadais (Analyst, 1909, **34**, 214), is accurate in the absence of tartrates, for which a preliminary qualitative test should be made. The modification of Gadais has the advantage of effecting complete precipitation by the addition of alcohol in such proportion that eventually a liquid is obtained containing 45 per cent. of alcohol, in which the citrate is insoluble. C. A. M.

Valuation of Fusel Oil. G. Heinzelmann. (*Zeitsch. Spiritusind.*, 1912, **35**, 612-613; through *J. Soc. Chem. Ind.*, 1912, **31**, 1142.)—Raw fusel oil should be bought on the basis of its content of amyl alcohols (b.-pt. 127° to 131° C.) which varies greatly in commercial samples. The author describes a method for determining the content of amyl alcohols by fractional distillation, used in the Institut für Gärungsgewerbe, Berlin. The best form of fractionating column for the purpose is that shown in the figure. The vapour rises in the enlarged portion of the column, outside the inner bulb-tube, and passes into the right-hand bulb, and thence through the spiral tube into the left-hand limb. The portion condensed in the latter limb runs down inside the bulb-tube, and forms a trap in the lower U-shaped portion, which should be at least 12 to 13 cms. long. When used with liquids which boil above 110° C., the column may be surrounded with cotton wool as far up as the spiral tube. The left-hand thermometer usually indicates 1·5° to 2° C. lower than the right-hand one. The method of fractionation employed is illustrated by the following example: 1,500 c.c. of raw fusel oil were distilled and collected in four fractions—viz., 540 c.c. passing over below 90° C., 420 c.c. between 90° and 115° C., 45 c.c. between 115° and 127° C., and 475 c.c. between 127° and 131° C. The first fraction, consisting chiefly of ethyl alcohol, but containing some water and fusel oil, was distilled from a water-bath (with the fractionating column) so long as ethyl alcohol passed over; the residue, which separated into two layers, one aqueous and the other fusel oil, was treated with sodium chloride (about 20 per cent. of the aqueous portion), and the fusel oil was isolated by means of a separating funnel. The second fraction, which consisted of two immiscible layers, aqueous and oily, was treated in the same way. The fusel oil thus obtained from the first two fractions was mixed and refractionated. When the thermometer indicated 112° C., the third fraction was added and distillation continued. The portion passing over above 127° C. amounted to 175 c.c., which, together with the fourth fraction from the original distillation (475 c.c.), gave a total yield of 650 c.c. of amyl alcohols, corresponding to 43·3 per cent. of the volume of the raw fusel oil. Particulars respecting three other samples of fusel oil are also given.



Interaction between Formaldehyde and Bismuth or Copper Compounds in the Presence of Alkali. F. Hartwagner. (*Zeitsch. anal. Chem.*, 1913, **52**, 17-20.)—When formaldehyde is added to bismuth hydroxide, reduction does not take place even when the mixture is heated; if, however, an excess of sodium hydroxide is also added, the whole of the bismuth is precipitated as metal. In the case of bismuth chloride, a large excess of both formaldehyde and sodium hydroxide must be added to insure the complete reduction of the bismuth. Copper may also be precipitated completely in the form of metal from its salts by the combined action of formaldehyde and alkali; it was observed that the finely-divided copper acted as a catalyst, and caused hydrogen to be evolved from the mixture of formaldehyde and alkali, these two substances reacting according to the equation:

$$HCOH + NaOH = HCOONa + H_2.$$

Further experiments showed that acetaldehyde in alkaline solution reduces gold and silver salts quantitatively; it also reduces gold salts in acid solution. Chloral hydrate reduces gold, silver, and copper salts completely in alkaline solution, but bismuth salts only partially. W. P. S.

Interaction between Formaldehyde and Nitrous Acid. L. Vanino and A. Schinner. (*Zeitsch. anal. Chem.*, 1913, **52**, 21-28.)—When an excess of formaldehyde is added to a nitrite solution and the mixture is acidified, the nitrite is decomposed according to the equation:

$$4HNO_2 + 3HCOH = 3CO_2 + 5H_2O + 2N_2.$$

This reaction forms a means for the estimation of nitrous acid. The nitrite solution is placed in a flask, a delivery-tube from which dips under the lower end of a measuring burette filled with potassium hydroxide solution. The air is expelled from the flask by boiling the solution; the flame is then removed, and clips on the delivery and inlet tubes of the flask are closed. Formaldehyde and hydrochloric acid are now drawn into the flask and the nitrogen evolved is collected in the burette, the last portion of the gas being expelled from the flask by boiling the contents until only steam and hydrochloric acid pass from the delivery-tube. The volume of the gas is then measured with the usual precautions, and its volume calculated into nitrous acid. This method cannot be employed, conversely, for the estimation of formaldehyde, as the reaction only takes place in the presence of an excess of the aldehyde. W. P. S.

Studies on the Chemistry of Gelatin and its Estimation. M. Berrár. (*Biochem. Zeitsch.*, 1912, **47**, 189-214.)—Water at a temperature of 21° C. dissolves 0·62 per cent. of gelatin; at 18° C., 0·60 per cent.; and at 17° C., 0·56 per cent.; the quantity of gelatin which is soluble in cold water is therefore approximately equal to the smallest amount of gelatin which is necessary to form a jelly. If pieces of gelatine be added to a saturated gelatin solution and the temperature is lowered, a portion of the dissolved gelatin separates out and no jelly is formed. Gelatin is not precipitated completely by alcohol or metaphosphoric acid, and an excess of the latter dissolves the precipitate first formed. The precipitate obtained when albumin is treated with metaphosphoric acid dissolves partially in an excess of the reagent. By precipitation with picric acid and potassium mercuric iodide, the authors find that the molecular weight of gelatin is 823. Gelatin is precipitated quantitatively from its aqueous solution by the addition of an equal volume of saturated picric acid solution, provided that the temperature of the mixture is below 8° C.; at higher temperatures even a large excess of picric acid does not precipitate the whole of the protein. The nitrogen in the precipitate may be estimated by Kjeldahl's method after reduction with iron turnings and acetic acid. The gelatin-picric acid compound dissolves in a 2 per cent. urea solution containing sodium chloride, and picric acid is therefore useless for the precipitation of gelatin in urine. Alcohol also dissolves the compound readily, and this forms a means of separating gelatin from albumin, albumoses, peptones, mucin, and casein, as the picric acid compounds of these substances are insoluble. For the estimation of gelatin in admixture with other proteins, it is recommended that an aqueous solution of the mixture be treated with a solution consisting of 1 part of saturated picric acid solution and 4 parts of alcohol; albumin, albumoses, peptones, mucin, and casein, are precipitated, and, after they have been removed by filtration, the gelatin is precipitated in the filtrate by cooling the latter below 8° C. and adding an excess of picric acid. This method may also be used for the detection of traces of gelatin in the presence of other proteins, as 1 part of gelatin per 100,000 parts of solution will yield a distinct turbidity on the addition of an excess of picric acid.

W. P. S.

Influence of Antiseptic Agents on the Reichert-Meissl Value of Fats. C. Grimaldi. (*Ann. Lab. Chim. Centr. delle Gabelle*, 1912, **6**, 631-640.)—An addition of benzoic acid to butter-fat, oleomargarine or cocoanut oil raises their Reichert-Meissl values, the increase being proportional to the amount added. In the usual proportions (0·1 to 0·2 per cent.) found in edible fats the influence of benzoic acid is insignificant. Salicylic acid also increases the Reichert-Meissl value of oleomargarine and cocoanut oil, but lowers the value of butter-fat when present in greater quantity than 0·5 per cent. In the usual proportion of about 0·1 per cent., salicylic acid has little appreciable influence upon the Reichert-Meissl value of any of the three fats. Attention is called to the fact that by the use of benzoic acid it is possible to prepare a mixture of fats giving the analytical values of genuine butter-fat. For example, a mixture of 78 per cent. of butter-fat (Reichert-Meissl value = 27·4), 9 per cent. of oleomargarine, 9 per cent. of cocoanut oil, and 4 per cent. of benzoic acid, gave the following results: Reichert-Meissl value, 26·1; saponification value, 230·7; refractive index at 40° C. (Zeiss), 44·1; and Maumené figure, 30·8. C. A. M.

Modification of Halphen's Test for Cottonseed Oil. E. Gastaldi. (*Ann. Lab. Chim. Centr. delle Gabelle*, 1912, **6**, 601-609.)—By substituting a small quantity of pyridin for the amyl alcohol in Halphen's reagent the test is rendered much more sensitive and the colorations more intense. A drop of pyridin is added to 5 c.c. of the oil under examination, and then 4 c.c. of carbon bisulphide containing 1 per cent. of sulphur in solution, and the tube heated in the water-bath for fifteen to thirty minutes. With an oil containing as little as 0·25 per cent. of cottonseed oil a yellowish-rose coloration is obtained, whilst Halphen's original reagent only gives a yellowish colour under the same conditions. The reaction is rendered more intense by heating the mixture in a closed vessel. As in the case of the original test, no reaction is obtained with cottonseed oil that has been heated to 250° C., or has had a prolonged treatment with sulphur dioxide. C. A. M.

Oxidation Assay of Essential Oils. F. D. Dodge. (*Eighth Int. Cong. App. Chem.*, 1912, vol. 6, 86-92.)—It is possible by selective oxidation with potassium permanganate solution to estimate the constituents in certain essential oils. For example, on chilling 10 c.c. of an oil containing eucalyptol (cineol) in a litre flask with a narrow neck, and adding saturated potassium permanganate solution in small portions (25 c.c.), so long as reduction takes place the eucalyptol is unaffected, whereas the other constituents of the oil are oxidised to soluble products. During the addition the flask is continually shaken and kept cool by means of ice, and when the liquid is no longer decolorised after standing for a few hours, the precipitated manganese oxide is dissolved by the addition of sulphurous acid solution (or sodium bisulphite and hydrochloric acid), care being taken to avoid an excess. The flask is then filled with water so as to bring the unoxidised oil into the neck, and this oil finally transferred by means of a capillary pipette into a graduated measuring-tube. In this way the eucalyptol in eucalyptus and cajeput oils may be quantitatively estimated. Rosemary oil yielded about 35 per cent. of unoxidised oil, containing, in addition to eucalyptol, camphor and bornyl acetate, which can be estimated in the usual manner. Spike (lavender) oil gave 41 per cent. of unoxidised oil consisting of eucalyptol and camphor. Test estimations with mixtures containing eucalyptol and terpenes showed that the latter were completely removed in the process. The fenchol in fennel oil is also unoxidised, but the separation is difficult owing to the formation of anisic acid (from the anethol) which separates as a crystalline deposit or is converted into manganese anisate. In the case of oils containing borneol or its esters, the latter are usually stable, whilst the free borneol is converted into camphor. The following amounts of unoxidised oil were separated: Pine needle (Siberian), 60; spruce, 45; sage, 16; and valerian (Japanese), 62 per cent. Menthol is readily oxidised to menthone, which is then slowly but completely oxidised. Menthyl acetate, however, is not attacked. All terpenes, with the exception of camphene, are rapidly oxidised. The slow rate at which camphene is oxidised enables it to be separated in this way in certain cases. Of other oils, cardamom left 8 per cent. (principally eucalyptol); pennyroyal, 5 to 10 per cent. (apparently menthone or a similar ketone); rue, 90 per cent.; cedar, 40 per cent.; copaiba (Para), 24 per cent. of unoxidised oil; and sandal (East Indian), a mixture of oil and a crystalline acid. Cymene is very slowly oxidised, and may be separated to a large extent from terpenes. The aldehydes of bitter almond and cassia oils are completely oxidised to benzoic acid, whilst cumin yields about 20 per cent. of cymene with terephthalic acid. In the case of phenols some yield simple oxidation compounds, whilst others form intermediate derivatives. Sassafras oil yields 5 per cent. of unoxidised oil containing camphor, and parsley and calamus oils from 5 to 6 per cent.

In applying the method to the detection of petroleum products in oil of turpentine, it was found that 10 c.c. of the latter require from 800 to 850 c.c. of the permanganate solution for oxidation. It is also possible to detect petroleum products in citronella oil in this way. The unoxidised part of the oil (which may normally contain camphor derived from the borneol) is treated with cold 80 per cent. sulphuric acid to eliminate the camphor, and the insoluble paraffins recognised by the fact of their not mixing with castor oil at 0° C. A further use of the process is to distinguish between bornyl acetate, which is not oxidised, and the isomeric linalyl, geranyl, and terpinyl acetates, which are readily oxidised. By estimating the actual amount of ester in an oil and that left after oxidation, the real amount of bornyl acetate—*e.g.*, in rosemary oil—is obtained. Fenchyl acetate, if present, would probably behave like bornyl acetate. C. A. M.

Studies on Fish Oils. Properties of Fish and Vegetable Oil Mixtures. G. F. White and A. Thomas. (*J. Ind. Eng. Chem.*, 1912, **4**, 878.)—The viscosity, fluidity, and density, through a range of temperatures, and the refractive index, and acid, saponification, and iodine (Hübl) numbers, of dogfish liver, soya bean, linseed, and China wood oils, and mixtures of these, were determined. The fluidities (the reciprocals of the viscosities) of fish and vegetable oil mixtures were found to be additive, except when either component decomposes on heating, as in the case of China wood oil; the viscosities are also additive if the viscosities of the components approximate each other closely. As has been shown already for fish oils, the fluidities are nearly proportional to the temperature. The fluidity of China wood oil is low, and is a criterion of its purity, as even small quantities of the other oils would raise its fluidity to an extent readily detected. The densities of the mixtures are approximately additive, and are directly proportional to the temperatures; China wood oil has a very high density. No general conclusions can be drawn from the refractive index, acid number, and saponification number. Linseed oil has a higher acid number than dogfish oil, and China wood oil a lower one. Small quantities of fish oil lower the refractive index of China wood oil. The iodine value is lowered by the addition of dogfish liver oil to vegetable oils. O. E. M.

Detection of Petroleum Products in Oil of Turpentine by Means of the Maumené Test. R. Massy. (*J. Pharm. Chim.*, 1912, **6**, 484-491.)—In Tortelli's method of testing oil of turpentine, 10 c.c. of the sample are treated in a Dewar's vacuum-tube with 10 c.c. of sulphuric acid (sp. gr. 1·722), the mixture stirred with a thermometer, and a note taken of the rise in temperature. Tortelli found that genuine commercial samples gave figures ranging from 100° to 105° C., and that in this way it was possible, not only to detect the presence of adulterants, but also to ascertain whether a pure oil had been altered by exposure to light and air. Oxidised oils gave a higher figure, the temperature rising more suddenly, whilst the residual mass in the tube was black and viscous instead of being only of a dark colour and fluid. In the author's experiments with oil of turpentine from *Pinus pinaster* (the only kind official in France), the Maumené figure with a pure oil was 96·5° C., whilst an addition of 10 per cent. of various petroleum products lowered the figure considerably. When other pure oils from *P. pinaster* were tested, however, results ranging from 93·2° to 106·0° C. were obtained. It would thus be possible to prepare mixtures of certain turpentine oils with 10 per cent. of white spirit giving figures well within these limits. To make the test reliable, the sample should be distilled so as to eliminate the bulk of the constituents other than pinenes, and the first 80 per cent. of the distillate collected. In the case of the oil from *P. pinaster* this distillate will consist of a mixture of α- and β-pinene in variable proportions, and will give fairly constant Maumené figures. Thus, fifteen samples gave results ranging from 96·2° to 100·6° C. A pure sample giving a value of 98·8° C. was mixed with 10 per cent. of various adulterants, and the distillate tested, with the following results: Turpentine oil with lamp oil, 91·3° C.; with rectified petroleum spirit, 84·6° C.; with ligroin, 82·2° C.; and with white spirit, 88·3° C. As little as 4 per cent. of white spirit was invariably detected in the test experiments. C. A. M.

Estimation of Sucrose in the Presence of Other Sugars. G. Testoni. (*Ann. Lab. Chim. Centr. delle Gabelle*, 1912, **6**, 582-589.)—The method devised by Margaillan (ANALYST, 1910, **35**, 315) gives good results with mixtures of pure lactose with other sugars, optically inactive lactic acid being formed, but is not satisfactory when applied to natural products, such as milk. When the Bulgarian bacillus is cultivated in milk, a mixture of dextro-rotatory and lævo-rotatory lactic acid is produced, in which the former predominates. The effect of this may be eliminated by removing the lactic acid by means of zinc sulphate. For example, in the case of condensed milk 20 grms. are dissolved in hot water, and the solution treated with acetic acid to coagulate proteins, then with basic lead acetate, followed by an excess of a saturated solution of sodium sulphate, and made up to 202 c.c., an allowance of 2 c.c. being made for the bulk of the coagulated compounds. The liquid is then filtered, and 100 c.c. of the filtrate treated with malto-peptone and 3 grms. of precipitated chalk, and the mixture neutralised, sterilised, infected with the Bulgarian bacillus, and incubated for six days at 35° C. Then, after concentration and cooling, the lactic acid is precipitated by the addition of a saturated solution of zinc sulphate, and the liquid made up to 100 c.c. and rapidly filtered. The sucrose may then be estimated polarimetrically in the filtrate. In a test experiment with a sample of condensed milk, the amount of sucrose thus found was 34·70 per cent. as against 34·84 per cent. found by Clerget's method. The alkali method of Jolles (ANALYST, 1911, **36**, 28) is shown to give good results with condensed milk. C. A. M.

Method for the Direct Polarimetric Determination of Sucrose in Presence of Certain Reducing Sugars. P. Lemeland. (*Zeitsch. Ver. deut. Zuckerind.*, 1912, 1192-1196; through *J. Soc. Chem. Ind.*, 1912, **31**, 1046.)—The optical rotatory and cupric-reducing powers of alkaline solutions of arabinose, dextrose, lævulose, galactose, and lactose are destroyed completely by heating with hydrogen peroxide in presence of a little manganese dioxide. Sucrose and dextrins are not affected by this treatment. For the determination of sucrose in presence of any of the reducing sugars mentioned above, a solution of the mixture is heated in a boiling water-bath with 0·5 grm. of manganese dioxide, or with 2 grms. if the quantity of reducing sugar present exceeds 10 grms. For every grm. of reducing sugars present, a mixture of 2 c.c. of 36 per cent. sodium hydroxide solution and

30 c.c. of hydrogen peroxide (German Officinal preparation) is added little by little to the hot liquid; this operation may be completed in thirty-five to forty-five minutes. The solution is then cooled, neutralised exactly with dilute acetic acid, decolorised with charcoal or basic lead acetate, made up to a convenient volume and polarised. The author cites a case in which a solution of 2 grms. of sucrose and 20 grms. of dextrose in 100 c.c. was treated with 2 grms. of manganese dioxide, 20 c.c. of sodium hydroxide, and 600 c.c. of hydrogen peroxide, as described. After cooling, nearly neutralising, concentrating to 400 c.c. on a water-bath, decolorising, and filtering, a polarimetric reading of +42′ was obtained instead of +40′ (control solution of sucrose alone). In analysing mixtures containing a high proportion of reducing sugars to sucrose, advantage may be taken of the solubility of sucrose in 91 per cent. alcohol, as in the following example: A mixture of 1 grm. of sucrose with 25 grms. of dextrose was left for twenty-four hours in contact with 100 c.c. of 91 per cent. alcohol and shaken frequently. The undissolved portion was separated by centrifuging, and washed twice with 50 c.c. of 91 per cent. alcohol. The combined alcoholic solutions were distilled, after addition of a trace of calcium carbonate, and the residual liquid was treated with the oxidising mixture as prescribed above. A polarimetric reading of +1° 14′ was obtained instead of +1° 18′ (control solution of pure sucrose). The new method cannot be applied to mixtures containing maltose, for this sugar is not completely decomposed by the oxidising mixture. Dextrins can be determined in presence of sugars other than maltose, any sucrose present being first inverted.

New Technical Determination of Bleaching Quality of Sulphite Pulp. E. Richter. (*Eighth Int. Cong. App. Chem.*, 1912, vol. 12, 233-236.)—The author had previously standardised Hempel's method for the estimation of lignin in cellulose pulps by heating with 13 per cent. nitric acid and measuring the gases produced, or titrating the nitrous acid with permanganate. He now describes a colorimetric method based on the same reaction, after having proved that the coloration produced is, under prescribed conditions, proportional to the percentage of residual lignin, and that this, again, is proportional to the consumption of bleaching-powder by the sulphite pulp under examination. Five grms. of the air-dry pulp are disintegrated, placed in a wide-necked bottle with 100 c.c. of 13 per cent. nitric acid, thoroughly shaken, and kept for about an hour in a dark place. After that time the bottle is again shaken, and the pulp is filtered off on a dry funnel with a plug of cotton-wool. Twenty-five c.c. of the filtrate are placed in a small bottle. A standard pulp of similar grade and of known bleach consumption is treated in the same manner. Water is added from a burette to the darker liquid until both solutions are identical in colour. Suitable amounts of each liquid are compared in Eggertz carbon tubes, looking down from above against white paper placed below, and using different amounts—5 c.c. and 10 c.c. Should the colours not be uniform, the shades may be adjusted by varying the depth of the column of liquid in one of the tubes. The bleach consumption of the test sample is then readily calculated by simple proportion compared with the standard sample. A very highly purified pulp may not produce sufficient coloration after one hour at the laboratory temperature; in such a case it is permissible to place the bottles in a water-bath at 40° C., at which temperature a coloration is developed in about fifteen minutes. J. F. B.

Estimation of the "Degree of Bleaching" [Chemical Condition] of Cotton. C. Piest. (*Zeitsch. angew. Chem.*, 1912, **25**, 2518-2522.)—In the nitrocellulose and artificial silk industries it is necessary to know the degree of chemical modification of the bleached cotton used as raw material. A number of empirical methods of analysis have been standardised by various authors, the majority of which are described in detail in Schwalbe's "Die Chemie der Cellulose." The author has carried out determinations of the more important of these "constants," first upon a series of cottons modified in known directions, and secondly upon a series of commercial bleached cotton wastes submitted for nitration purposes. The principal "constants" studied were—(1) The wood-gum value—*i.e.*, the constituents soluble in cold 5 per cent. sodium hydroxide, and reprecipitated on acidification and addition of alcohol; (2) Schwalbe's total copper value (boiling Fehling's solution); (3) the cupric hydroxide value—*i.e.*, the amount of copper absorbed from cold Fehling's solution; (4) Vieweg's acid value—*i.e.*, the amount of sodium hydroxide neutralised on boiling for fifteen minutes with a 1 per cent. solution; (5) the amount of cupric hydroxide absorbed from a 10 per cent. solution of copper sulphate; (6) Ost's viscosity value, representing the viscosity of solutions of the cotton in cuprammonium solution prepared under rigidly prescribed conditions. The results obtained with cottons specially modified are summarised in the following table:

Nature of Cotton.	Schwalbe's Copper Value.	Cupric Hydroxide Value.	Vieweg's Acid Value.	Copper Sulphate Value.	Ost's Viscosity Test. Solution diluted	
					After 24 Hours.	After 48 Hours.
1. Normally prepared nitrating cotton	1·64	0·53	1·86	2·58	26·50	17·35
	1·64	0·57	1·86		24·40	16·50
					22·00	15·77
2. Overbleached cotton	8·76	0·25	6·48	1·54	2·70	2·58
	8·76	0·25	6·40		2·70	2·58
					2·70	2·58
3. Very strongly oxidised cotton	16·50	0·52		1·08	2·23	2·12
	16·00				2·23	2·12
					2·23	2·12
4. Sample No. 2 digested for twenty-four hours with cold 5 per cent. sodium hydroxide	3·79		4·30	2·97	2·64	2·58
	3·79		3·65		2·64	2·58
			3·86		2·64	2·58
5. Mercerised cotton	1·52	1·47	0·40	1·87	17·46	14·15
	1·52	1·47		1·91	16·15	13·45
					14·62	12·88
6. Hydrocellulose prepared with sulphuric acid, sp. gr. 1·53 in the cold	4·13	traces	7·04	2·33	2·65	2·43
	4·13		7·04	2·33	2·65	2·43
					2·65	2·43

The above figures, in conjunction with others obtained with commercial samples of normally bleached nitrating cottons, lead to the following conclusions: Normally bleached cotton should show a "wood-gum" value not exceeding 1; the total copper value should not exceed about 1·5, and preferably should not be higher than 1; from this is to be deducted the cupric hydroxide value, which, except for mercerised cotton, should range round about 0·5. Vieweg's acid value, which is a measure of the constituents readily susceptible to hydrolysis by alkalis, should be considerably less than 2, preferably less than 1·5. Ost's viscosity value is an extremely delicate test for chemical condition; this value decreases the longer the solution is allowed to stand before dilution prior to making the observation. Moreover, in the case of normal cottons, when three estimations are made consecutively, each observation is lower than the previous one. The author considers that normal cottons should show a viscosity of at least 10 when the solution is allowed to remain for five days before dilution; this period is selected because the differences between three consecutive observations are then less pronounced. Modification by overbleaching (oxidation) is shown by an abnormally high copper value, a low or normal hydroxide value, a high acid value according to Vieweg, and a low viscosity. Extraction of the constituents of the oxycellulose soluble in alkali lowers the copper value, which, however, still remains abnormal. Vieweg's acid value is changed in a similar manner, but the viscosity value is unchanged. Mercerisation increases the cupric hydroxide value, which is a specific criterion of hydrated cellulose; Vieweg's acid value is lowered owing to the elimination of hydrolysable constituents by the strong mercerising lye, whilst the viscosity value is not appreciably lowered by the short action of mercerising alkali. Modification by the action of acids (hydrocellulose) results in an increase of the copper value, but in less degree than modification by oxidising agents; the cupric hydroxide value is lowered, but the sensitiveness to alkaline hydrolysis (acid value) is very high in relation to the copper value; the viscosity is lowered, just as with overbleached cellulose. The author considers that the copper sulphate value is of subordinate importance as a diagnostic test.

J. F. B.

Researches on the Gelatinising Temperatures of Starch Granules.

M. Nyman. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1912, **24**, 673-676.)—In a first series of experiments various starches were mixed with water heated to a suitable temperature, and as the temperature was raised drops of the mixtures were removed every thirty seconds and examined under the micro-polariscope. The point at which the granules ceased to show interference crosses was taken as the gelatinising temperature. Rye, barley, and wheat starches were found to gelatinise at slightly different temperatures, the results obtained being—Rye starch, 57° C.; barley starch, 58° C.; and wheat starch, 59° C. Further experiments on the rate at which the gelatinisation proceeded showed that the larger starch granules gelatinised more quickly than the smaller. Although the differences in the gelatinising temperatures does not afford a means of identifying two starches in a mixture of the same, the rate of gelatinisation may be of some use for the purpose. If a mixture of rye and wheat meals is maintained at a temperature of 53° C., the rye starch is gelatinised in six minutes, whilst the wheat starch requires twenty-four minutes; any ungelatinised starch granules observable in such a mixture after seven minutes' heating indicate the probable presence of wheatmeal. W. P. S.

INORGANIC ANALYSIS.

Method for the Analysis of Barium Sulphide. J. F. Sacher. (*Zeitsch. anal. Chem.*, 1913, **52**, 28-31.)—A definite volume of the barium sulphide solution is treated with an excess of standardised lead nitrate solution, the mixture is rendered slightly acid with acetic acid, and after the lapse of a short time the lead sulphide is separated by filtration and washed with cold water. The excess of lead in the filtrate and wash-water is then titrated with ammonium molybdate solution, as described previously by the author (*cf.* ANALYST, 1910, **35**, 79). W. P. S.

Estimation of Small Quantities of Carbon Monoxide. O. Brunck. (*Zeitsch. angew. Chem.*, 1912, **25**, 2479-2481.)—The method described depends on the reduction of palladium chloride to metallic palladium by the action of carbon monoxide. The gas in which the carbon monoxide is to be estimated is collected in a flask of from 0·5 to 1 litre capacity; this flask is fitted with a rubber stopper having two holes, which are closed by means of two short pieces of glass rod, and the actual capacity of the flask is determined accurately. After the flask has been filled with the gas, about 20 c.c. of a sodium-palladous chloride solution (containing a quantity of the salt per litre equivalent to about 4·7 grms. of palladium) and 10 c.c. of a 5 per cent. sodium acetate solution are added successively by means of a pipette the stem of which passes through one of the holes in the stopper; while the solutions are being added the glass-rod in the other hole is loosened slightly. The glass rods are at once reinserted in the stopper, and the flask is agitated gently for about thirty minutes. Should the liquid in the flask remain quite clear, carbon monoxide is not present; but if metallic palladium is precipitated it is collected on a filter, washed with hot water, incinerated, and the metal heated in a current of hydrogen before being weighed. One grm. of palladium is equivalent to 0·2624 grm. or 210 c.c. (N.T.P.) of carbon monoxide. It has already been proposed to absorb carbon monoxide in cuprous chloride solution, and to then add palladous chloride, but Winkler has shown that the metallic palladium precipitated under these conditions always retains copper, even after washing with hydrochloric acid and ammonia. W. P. S.

Estimation of Copper by Means of Sodium Hypophosphite. R. Windisch. (*Zeitsch. anal. Chem.*, 1913, **52**, 1-13.)—The method described by Dallimore (*cf.* ANALYST, 1909, **34**, 452) was found to yield accurate results; pure copper sulphate yielded 31·60 per cent. of cupric oxide against 31·78 per cent. required by theory and 31·62 found by the electrolytic method. The author prefers to use sodium hypophosphite in place of hypophosphorus acid as recommended by Dallimore. A quantity of about 100 grms. of the copper salt under examination are dissolved in water, and diluted to 1 litre; 50 c.c. of this solution are acidified with 3 c.c. of sulphuric acid, 20 c.c. of a 20 per cent. sodium hypophosphite solution are added, and the mixture is heated to boiling for ten minutes. The reduced copper is then collected on a filter, dried, ignited to oxide, and weighed. The presence of considerable amounts of iron salts does not affect the accuracy of the results obtained by the method. W. P. S.

Analysis of Ferro-Silicon. S. Camilla and C. Pertusi. (*Ann. Lab. Chim. Centr. delle Gabelle*, 1912, **6**, 627-630.)—Since the impurities in commercial ferrosilicon (aluminium, manganese, phosphorus, sulphur, and carbides) never exceed 2 per cent., the following rapid method of analysis will give results sufficiently accurate for general purposes: From 0·4 to 0·5 grm. of the finely powdered sample is treated in a platinum crucible with 20 to 30 c.c. of hydrofluoric acid, followed by a few drops of concentrated nitric acid. As soon as a clear solution is obtained, a few c.c. of dilute sulphuric acid are added, the liquid evaporated to dryness, and the residue ignited in a muffle until constant in weight, and weighed as ferric oxide. The silicon is then found by difference. C. A. M.

Influence of Colloids on the Electrolytic Separation of Lead. H. Freundlich and J. Fischer. (*Zeitsch. Elektrochem.*, 1912, **18**, 885-891; through *Chem. Zentralbl.*, 1912, II., 1885.)—It is known that the cathodic deposition of certain metals, particularly lead, is assisted by the presence of certain foreign matters of colloidal nature. The deposition itself is probably a complex process, and consists of discharging, dehydration, and crystallisation. It would, then, appear that the action of the colloid consists in a retardation of the crystallisation velocity, as has recently been suggested by Marc. In that case a parallelism should exist between the protective action of the colloid, for instance, towards colloidal gold solution and its power of facilitating the cathodic deposition of lead. The author has tested this hypothesis experimentally. To solutions of lead in hydrofluosilicic acid and perchloric acid there were added during electrolysis increasing quantities of gelatin, dextrin, and a few other organic colloids, and the quantities necessary for the production of a smooth, adherent deposit of lead were estimated. Then the protective action of the same colloids in gold solutions was tested, and the quantities were estimated which were required to change the red solution into violet. No definite parallelism which would confirm the above hypothesis could be observed. Nevertheless, the investigations indicated that the colloids are adsorbed by nascent germs of the metal, that the crystallisation velocity is thereby retarded, and the compactness of the deposit improved; also in the precipitation of the lead by zinc a similar influence of colloids was established as in the case of electrolysis.

J. F. B.

Calorimetric Detection and Estimation of Magnesium Oxide in Magnesium Carbonate, Asbestos Mixtures, etc. V. Fortini. (*Ann. Lab. Chim. Centr. delle Gabelle*, 1912, **6**, 509-512.)—Magnesium oxide (or other oxide of an alkali earth) may be estimated in carbonates or mixtures containing asbestos, glass wool, etc., by the rise of temperature produced on treatment with dilute hydrochloric acid. Twenty-five c.c. of the acid (sp. gr. 1·19 diluted with an equal quantity of water) are placed in a small Dewar's vacuum tube, the temperature taken, a weighed quantity (0·5 grm. or 1 grm.) of the substance introduced, the mixture stirred with the thermometer, and the rise in temperature noted. The increase will be proportional to the quantity of magnesium oxide, etc., in the sample, provided that the same calorimeter and method of working be used. The apparatus is first standardised upon pure magnesium oxide and calcium oxide. For example, in the author's experiments the following results were obtained:

Magnesium oxide (grms.) ...	1·0	0·5	0·4	0·3	0·2	0·1
Rise in temperature (° C.)	37·0	19·0	13·9	11·3	7·3	3·7
Calcium oxide (grms.) ...	1·0	0·5	0·4	0·3	0·2	0·1
Rise in temperature (° C.)	36·8	18·8	14·8	11·4	7·4	3·7

No rise of temperature was obtained with pure neutral magnesium carbonate or calcium carbonate, but ordinary commercial magnesium carbonate showed a rise of 1° to 2° C. per grm., owing to the presence of small quantities of basic carbonate.

In the examination of mixtures containing magnesium oxide the following results are typical of those obtained. In each case 1 grm. was used.

Magnesium Oxide.	Magnesium Carbonate.	Asbestos.	Increase of Temperature.	
			Observed.	Calculated.
Per Cent.	Per Cent.	Per Cent.	° C.	° C.
50	50	—	19·0	18·5
60	40	—	14·8	14·8
70	30	—	11·5	11·1
80	20	—	7·2	7·4
90	10	—	3·8	3·7
10	40	50	3·4	3·7
20	30	50	7·1	7·4
20	40	40	7·2	7·4
40	30	30	14·3	14·8
20	50	50	7·2	7·4

C. A. M.

Reduction of Manganese Dioxide by Nitrous Acid, and the Use of this Reaction in the Estimation of Phosphorus in Iron and Steel without Separation of Silicon. E. R. E. Müller.

(*Chem. Zeit.*, 1912, **36**, 1490.)—In the author's method for estimating phosphorus without the separation of silicon (*Chem. Zeit.*, 1911, **35**, 1201) sodium peroxide was used to reduce the manganese dioxide formed. The following procedure may be substituted with advantage: After the boiling with permanganate (2 c.c. of a solution containing 40 grms. per litre), the beaker is withdrawn from the hot-plate, and 0·5 c.c. of absolute alcohol and 1 c.c. of 10 per cent. sodium nitrite solution added. On agitation the manganese dioxide is dissolved, and evolution of ethyl nitrite usually begins; 1 c.c. of concentrated ammonia is added, and the solution precipitated with ammonium molybdate solution.

Not more than 0·5 c.c. of alcohol should be used, as otherwise the quantity of nitric acid present is unduly reduced. The sodium nitrite is added to start the reaction.

O. E. M.

Qualitative Detection of Nitric Acid in Sulphuric Anhydride. W. N. Iwanow. (*J. Russ. Phys. Chem. Soc.*, 1912, **44**, 1334-1337; through *Chem. Zentralbl.*, 1912, II., 1944.)—In testing sulphuric anhydride for the presence of nitric acid by the diphenylamine test erroneous conclusions may be drawn in certain cases. A sulphuric anhydride containing 20 per cent. of trioxide and free from nitric acid gives no coloration with diphenylamine, but a product containing more than 20 per cent. of trioxide may sometimes give a coloration owing to the fact that the sulphur trioxide may act as an oxidising agent, splitting up into sulphur dioxide and active oxygen. For testing acids containing much sulphur trioxide, the author recommends that 10 c.c. of the fuming acid, containing about 30 per cent. of trioxide, should be diluted with 20 c.c. of pure sulphuric acid of sp. gr. 1·84, so that the mixture represents approximately a monohydrate. The mixture is cooled, treated with 1 c.c. of the diphenylamine reagent, shaken, and allowed to stand for a minute; if no blue coloration appears, the acid is free from nitric acid.

J. F. B.

Osmium Tetroxide as an Oxygen-Carrier and the Rendering Active of Chlorate Solutions. K. A. Hofmann. (*Ber.*, 1912, **45**, 3329-3336.)—The reaction of osmium tetroxide with fats is known to be one of oxidation, the black coloration being that of the reduced osmium dioxide formed. In order to utilise this property of osmium tetroxide for practical purposes it is desirable to make the reaction catalytic by ensuring the reoxidation of the dioxide to the tetroxide as fast as the latter is deoxidised. The action of atmospheric oxygen in this connection is distinct, but feeble and slow; it can be demonstrated by shaking acid solutions of leuco-bases with air and a trace of the tetroxide. Oxidising effects which are satisfactory and rapid may, however, be carried out with oxygen under a pressure of 50 atmospheres at temperatures of 50° to 100° C. in the autoclave, using osmium tetroxide as a catalyst in the proportion of 0·01 grm. per 200 c.c. of liquid. Ethyl and methyl alcohols in aqueous solution give, in this manner, mixtures of the corresponding aldehydes and acids. Aldehydes are comparatively resistant under these conditions; anthracene in acetic acid solution is oxidised to anthraquinone. The most generally effective oxidising agent for preparation purposes consists of a neutral or faintly acid solution of a chlorate rendered active by a trace of osmium tetroxide. For instance, a mixture of 15 grms. of arsenious acid, 10 grms. of potassium chlorate, and 50 c.c. of water shows no reaction even after the addition of a little dilute sulphuric acid; but if 1 c.c. of a solution of 1·5 grm. of osmium tetroxide in 100 c.c. of water be added, a violent oxidation ensues with rapid rise of temperature. A large number of analogous oxidations of inorganic and organic substances are described. The reaction may be utilised for the estimation of hydrazine; for instance: 0·02288 grm. of hydrazine sulphate with 50 c.c. of water and 2 grms. of potassium chlorate, when treated with 1 c.c. of the osmium solution in a current of carbon dioxide, gave 43·64 c.c. of nitrogen, equal to 21·59 per cent., as against 21·55 per cent. calculated.

Rapid oxidations of indigo carmine, quinol, aniline, benzidine, pyrocatechol, tannic acid, resorcinol, anthracene, and other substances are described. Quantitative experiments with alcohol proved that the reaction is purely one of oxidation with no tendency to chlorination. The plain solution of osmium tetroxide may be used as a test for pure benzene, which shows no blackening, whilst a trace of thiophene impurity reduces the tetroxide to the black dioxide. Pure pentane and hexane also do not reduce, whereas the commercial hydrocarbons and those with ethylene bonds reduce readily. Saturated ketones are not affected, but commercial acetone shows the black coloration. J. F. B.

Estimation of Available Oxygen in Per-Salts by Means of Titanium Trichloride. L. Moser and F. Seeling. (*Zeitsch. anal. Chem.*, 1913, **52**, 79-86.)—The method depends on the reaction between titanium trichloride and the hydrogen peroxide which is formed when perborates, percarbonates, etc., are treated with water; the reaction proceeds according to the equations:

$$NaBO_3 + H_2O = NaBO_2 + H_2O_2 \text{ and } H_2O_2 + 2TiCl_3 + 2HCl = 2TiCl_4 2H_2O.$$

Pertitanic acid is first formed, and produces a yellow coloration in the solution; this acid is decomposed on the addition of a further quantity of titanium trichloride, and the disappearance of the yellow colour is taken as the end-point of the titration. A weighed quantity of perborate is dissolved in cold water in a flask, dilute sulphuric acid is added, and the mixture is titrated with a standardised titanium trichloride solution, while an atmosphere of carbon dioxide is maintained in the flask. The process may be applied to washing powders containing perborates, but when soap is present it is advisable to add about 10 c.c. of carbon tetrachloride after the addition of the sulphuric acid, in order to remove the fatty acids from the sphere of action of the pertitanic acid. In the case of percarbonates the sample must be dissolved in ice-cold water, and an aliquot portion of the solution then pipetted into an excess of the titanium trichloride solution; the excess of the latter is titrated with ferric sulphate solution. A current of carbon dioxide is passed through the flask during these operations. In the case of ammonium persulphate there is no formation of pertitanic acid; a solution of the salt is therefore treated with an excess of titanium trichloride, and the mixture then titrated with ferric sulphate solution, using potassium thiocyanate as indicator. W. P. S.

Estimation of Palladium, and its Separation from Copper and Iron. M. Wunder and V. Thüringer. (*Zeitsch. anal. Chem.*, 1913, **52**, 101-104.)—Palladium forms a compound with dimethylglyoxime which is practically insoluble in dilute acids (the solubility in 2 per cent. hydrochloric acid is 0·001 per cent.); the compound is, however, readily soluble in ammonia and potassium cyanide solution, and in this respect differs from the corresponding nickel compound (*cf.* ANALYST, 1909, **34**, 74). The palladium compound contains 31·61 per cent. of the metal. For the estimation, the palladium solution is acidified with hydrochloric acid, diluted to 150 c.c., and an excess of a 1 per cent. dimethylglyoxime solution (in hydrochloric acid) is added. The mixture is heated for thirty minutes on a water-bath, and the precipitate is then collected on a filter, washed, dried, and ignited. A very small flame must be used at the commencement of the ignition in order to prevent loss of metal, but when the filter is incinerated completely the heat may be increased to bright redness. When the solution contains copper and iron in addition to palladium, the latter is first separated as described above, but a larger proportion of the reagent is required should ferric salts be present. The filtrate from the palladium compound is treated with sulphurous acid and potassium thiocyanate, in order to precipitate the copper, and the iron is then separated as sulphide, dissolved in hydrochloric acid, oxidised, and precipitated as hydroxide.

W P. S.

Detection and Estimation of White Phosphorus in Phosphorus Sesquisulphide. T. Schloesing jun. (*Comptes rend.*, 1912, **155**, 1461-1463.)—The method is based upon the difference in solubility of white phosphorus and phosphorus sesquisulphide in that fraction of petroleum spirit distilling below 45° C. Twenty grms. of the sample are shaken vigorously for some minutes with 125 c.c. of the solvent, and if it does not immediately clarify, the liquid is rapidly filtered through asbestos, with precautions against loss by evaporation. An aliquot portion (100 c.c.) of the filtrate is transferred to a tared flask, immersed in water at 15° to 20° C. and evaporated with the aid of a water-pump, and traces of solvent expelled by a current of air, after which the residue is weighed. In the case of pure sesquisulphide the ratio of phosphorus to sulphur will approximate $\frac{P_4}{S_8} = \frac{56{\cdot}4}{43{\cdot}6}$. In this way the following results, calculated upon the total 125 c.c., were obtained with pure phosphorus sesquisulphide alone and with the addition of 1 per cent. of white phosphorus :

		A. Pure Sesquisulphide.	B. With 1 per Cent. of Phosphorus.
Residue (mgrms.)		284	433
Excess over 284 mgrms. ...		—	149
Composition of residue $\left.\begin{matrix}P=164\\S=131\end{matrix}\right\}$		295	$\left\{\begin{matrix}300\\143\end{matrix}\right\}$ 443
Ratio... ... ... $\frac{P}{S}\,\frac{164}{131}$	=	$\frac{55{\cdot}6}{44{\cdot}4}$	—

The differences between the sums of the phosphorus and sulphur and the residues obtained were attributed to errors of manipulation. The white phosphorus added to B was not entirely recovered, a portion having combined with the sesquisulphide to form a sulphide only a fraction of which was dissolved. The amount of phosphorus remaining in the free state was calculated from these results to be 120 mgrms., or 0·6 per cent. Phosphorous and phosphoric acids, which are frequently present in commercial sesquisulphide, have no influence upon the results. The solubility of white phosphorus in the rectified petroleum spirit used was 8 grms. per litre. The phosphorescence shown by sesquisulphide containing a little free white phosphorus is greatly intensified by applying the test to the residue from the petroleum spirit. A little fine quartz sand is added to the petroleum spirit solution before evaporation *in vacuo*, and the last traces of solvent expelled with air. On now shaking the sand in the flask in the dark, phosphorescence is observed in the presence of minute traces of white phosphorus. C. A. M.

Estimation of Potassium by Cobaltinitrite Method. F. H. MacDougall.

(*J. Amer. Chem. Soc.*, 1912, **34**, 1684-1686.)—In the usual method the yellow cobaltinitrite compound, $K_2NaCo(NO_2)_6,xH_2O$, is boiled with excess of $\frac{N}{10}$ permanganate for several minutes, dilute sulphuric acid is added, then oxalic acid, and finally $\frac{N}{10}$ permanganate till the end-point is reached. Thus the oxidation of the nitrite takes place in alkaline solution, and the factor for the $\frac{N}{10}$ permanganate is 0·00071 grm. K per 1 c.c. The author now proposes to oxidise the nitrite in acid solution, adding first dilute sulphuric acid to the yellow precipitate, and finally oxidising with permanganate, when 1 c.c. = 0·000651 grm. K. (In acid solution the cobaltic salt formed changes to the cobaltous salt with evolution of oxygen, and thus in the acid method the amount of permanganate required is greater than in the alkaline method in the proportion of 12 to 11.) The results compare favourably with the older method. A. R. T.

Determination of Chromium and Vanadium in Steels. D. J. Davenport.

(*J. Ind. Eng. Chem.*, 1912, **4**, 895.)—The author's method for the estimation of vanadium (*J. Ind. Eng. Chem.*, 1912, **4**, 249) is extended to steels containing chromium in addition. To a solution of 2 grms. of the sample in 12 c.c. of strong sulphuric acid and 50 c.c. of water are cautiously added 25 c.c. of nitric acid (sp. gr. 1·42), oxides of nitrogen removed by boiling, and sodium bismuthate added until the permanganic acid formed from the manganese present does not disappear on shaking. The solution is diluted to 200 c.c., and, after addition of a further small quantity of sodium bismuthate, boiled for twenty minutes to convert the permanganic acid into manganese dioxide. It is cooled by the addition of 50 c.c. of water, filtered through asbestos, made up to 300 c.c., cooled, 5 c.c. of syrupy phosphoric acid added, and $\frac{N}{100}$ ferrous sulphate solution run in until there is an excess, as shown by testing on a plate with ferricyanide; an excess of several c.c. over the amount of $\frac{N}{100}$ potassium permanganate required to produce a permanent pink tint is added, the solution stirred for one minute, and the pink colour just removed with $\frac{N}{100}$ ferrous sulphate solution. The total ferrous sulphate less the permanganate, multiplied by 0·000173, gives the amount of chromium present. To determine the vanadium, ferrous sulphate considerably in excess of that required to reduce the vanadium and 1 grm. of powdered manganese dioxide are added in such a fine state of division that it will settle clear in fifteen seconds in a beaker of water. The solution is shaken until all ferrous iron is oxidised to ferric, as shown by the ferricyanide test, and filtered with suction through asbestos; $\frac{N}{100}$ potassium permanganate is run in until a permanent pink is reached, when several c.c. in excess are added and the solution is shaken for one minute. The pink tint is then just removed with $\frac{N}{100}$ ferrous sulphate solution. The permanganate minus the ferrous sulphate, multiplied by 0·00051, gives the vanadium present.

O. E. M.

Direct Volumetric Determination of Tin. H. B. J. Rawlins. (*J. Soc. Chem. Ind.*, 1913, **32**, 27.)—In an investigation to ascertain the degree of accuracy attainable by technical methods in the direct determination of tin in the commercial metal, the following processes were employed: (1) Titration with excess of ferric chloride and working back with a dilute solution of titanium chloride (method of Knecht and Hibbert, *J. Soc. Chem. Ind.*, 1905, **24**, 1192); (2) titration with ferric chloride, employing starch and cuprous iodide as indicator; (3) titration with iodine. The procedure adopted in the second and third methods consisted in oxidising the bulk of the stannous chloride by the addition of a definite volume of a strong solution of ferric chloride or iodine from an automatic burette, and completing the titration with a corresponding weak solution. It was found that by treating 1 grm. of the metal (thinly rolled or in the form of filings) with 50 c.c. of cold or slightly warm strong hydrochloric acid in a flask through which a current of carbon dioxide was maintained, and into which a small piece of marble had been previously placed, solution was effected in from forty-five to ninety minutes, the whole of the tin being retained in the stannous condition. In determinations made by the three methods upon commercial tin containing only traces of antimony, the maximum differences obtained (in terms of tin) were 0·14 per cent. by the first, 0·15 by the second, and 0·10 by the third process. Copper in small quantity was found to be practically without influence on the titanium chloride titration; it is generally known, however, that finely divided antimony is dissolved by solutions of ferric chloride and iodine, but since this constituent is present in only the smallest quantity in many brands of commercial tin—and when present may be determined and allowed for—the author considers that the results for tin obtainable by the direct methods indicated are not only sufficiently accurate for technical purposes, but in many cases more correct than those obtainable by the "difference" method involving rapid technical determinations of the impurities.

Estimation of Titanium in the Presence of Iron. W. J. Thornton jun. (*Zeitsch. anorg. Chem.*, 1912, **79**, 190-194.)—The following rapid modification of Gooch's method of estimating titanium in the presence of iron is applicable in the case of minerals containing but little aluminium. Iron is first removed by adding tartaric acid (three times the weight of the total oxides), introducing hydrogen sulphide until the iron is converted into the ferrous state, then adding a slight excess of ammonia, and continuing the current of hydrogen sulphide until the iron is completely precipitated, leaving the liquid still faintly alkaline to litmus. The precipitate is separated and washed with very dilute ammonium sulphide solution, and the filtrate and washings will then contain the whole of the titanium. The tartaric acid in solution must next be destroyed, and this is best done by means of a mixture of sulphuric and concentrated nitric acid. For example, to 25 c.c. of a solution containing about 0·1 grm. of titanium oxide there are added from 10 to 12 c.c. of concentrated sulphuric acid, and the liquid is evaporated until the tartaric acid begins to carbonise. The dish is then covered with a clock-glass, and the heating continued until frothing begins, after which its contents are cooled and cautiously treated with 5 c.c. of concentrated nitric acid. The pale yellow viscous fluid eventually obtained is poured into 100 c.c. of cold water, and the titanium precipitated by the basic acetate method. A single precipitation is sufficient when only a little aluminium is present, and in this way the original method is greatly accelerated.

C. A. M.

Present Position and Importance of Analytical Chemistry. W. Fresenius. (*Zeitsch. angew. Chem.*, 1912, **25**, 2513-2518.)—Reviewing the development of analytical chemistry, the author states that this branch of the science, though merely a means to an end, has become a factor of indispensable importance in most departments of public activity, comparable in its fundamental utility with the art of calculating. In the domain of pure science, also, the fixation of the atomic weights, the periodic law, the phenomena of isomorphism, isomerism, and homologous series, are founded on the results of chemical analysis. The reawakening of interest in inorganic and physical chemistry, which may be dated from about 1880, has led to a complete change of attitude in the scientific treatment of analytical chemistry. In this connection may be cited the recognition of reversibility of reactions and the laws which govern it, the recognition of the influence of the size of particles on their solubility, the conceptions of occlusion and adsorption, the ionisation of electrolytes. In volumetric analysis many reactions, which do not involve the removal of one of the components in the form of a precipitate or a gas, have been recognised as complex equilibria, the results of which do not follow the simple chemical equations, but depend on the total factors present; thus, it is commonly found that volumetric solutions can only be standardised against substances similar in nature to the material analysed. The application of physical methods has attained a great development, and the principles involved in the standardisation and use of "constants" for analytical purposes have opened up new fields. The ionic theory has revolutionised the previous conceptions of acidimetry, and the analyst now has to take into account the temporary ionic conditions of the liquid to be titrated, often disregarding the total potential acidity as revealed by ordinary titration. Microscopic analysis, starting from petrography, has spread to metallurgy and other fields of usefulness.

J. F. B.

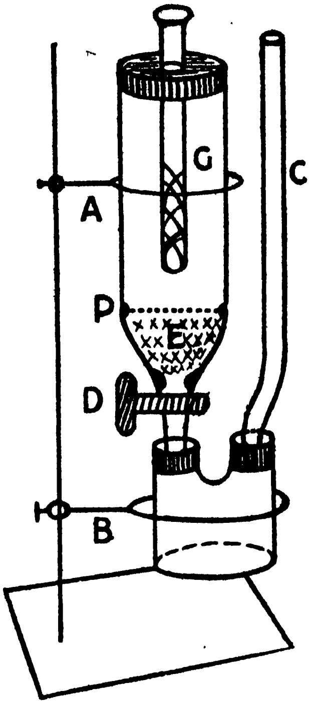
Microchemical Reagents. W. Lenz. (*Zeitsch. anal. Chem.*, 1913, **52**, 90-99.) —Directions are given for the preparation and storing of reagents used in microchemical analysis. Glass vessels must be avoided, as they yield soluble substances to acids, etc.; for instance, if a drop of sulphuric acid which has been kept in a glass bottle is evaporated on a microscope slide, a distinct residue will be obtained in which potassium and sodium may be detected microchemically. It is recommended that all acids, ammonia, and solid reagents, be stored in small silica bottles, and that water (freed from ammonia) be stored in a gilded silver flask. The liquid acids and ammonia must be distilled from silica flasks, the condenser and receiver also being made of silica. Microscope slides made of silica should be used in microchemical analysis.

W. P. S.

APPARATUS, ETC.

Estimation of Solubilities. W. R. Forbes. (*Chem. News*, 1912, **106**, 300.)—The author has modified Goldschmidt's apparatus (see figure) by widening the filter area, eliminating rubber connections, and simplifying the stirrer (*G*). Water and the salt are placed in *A*, and the tap *D* closed. When the solution is saturated, *D* is opened, and the liquid drawn into the weighing bottle *B* by exhausting through *C*. The weighing bottle is detached from its ground-glass joints with *A* and *C*, dried and weighed. When in use the apparatus and stand are placed in a water-bath at the desired temperature.

A. R. T.



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REVIEWS.

General Chemistry of the Enzymes. Hans Euler. Translated from the revised and enlarged German edition by Thomas H. Pope. New York: John Wiley and Sons. London: Chapman and Hall, Ltd. 1912. Pp. 311. Price 12s. 6d. net.

The author in this work states that he does not claim to give a complete synopsis of our knowledge of the enzymes, but only to review the more important facts from a general standpoint, and to fit them, so far as is possible, into their proper places in the fabric of general and physical chemistry. The volume is based on two reports written by the author for the "Ergebnisse der Physiologie" in 1907 and 1910, and he expresses the hope that the present monograph, which is intended more especially as an aid to scientific research, will also prove useful to those concerned with the practical applications of enzymic actions. Many students of this branch of chemistry will echo the question of the author, and ask whether the time is yet ripe for giving a representation of the physical chemistry of the enzymes, but after a perusal of the present laborious undertaking, they will probably agree with him that the attempt should be made, especially as the results of many of the more recent and exact investigations correspond more closely with those required to satisfy physico-chemical theories.

The modern technical methods of .measuring enzymic activity are daily becoming more numerous and important, as the ever-growing and highly specialised literature of the subject bears witness, and it would surprise many not familiar with recent research to learn the high degree of accuracy in conjunction with the most refined methods which are now employed in the assay and standardisation of such preparations as commercial pepsin, rennin, or diastase. Sörenson and many others have emphasised the extreme importance of precision in the technique of such work, and essential details, such as the exact reaction of the media employed and other points previously too often overlooked, now receive due attention from all careful workers.

Following a chapter on the special chemistry of the enzymes is an account of their physical properties, so far as they are known, and of the "activators (co-enzymes), paralysors, and poisons" known to affect their actions. The longest section of the book, and that to which most attention has evidently been given, deals with the Chemical Dynamics of Enzyme Reactions, and the work of a host of investigators in this much-worked field is in the main ably summarised. The author would appear, however, to have missed the significance of an important paper by Adrian Brown, to which insufficient justice is done on p. 162, but rightly points out that some of the recorded experimental work (notably some of Henri's) is vitiated from the fact that when optical methods were employed for measuring such reactions as the inversion of cane-sugar, sufficient care was not taken to obviate the disturbing effects of bi-rotation.

Short chapters on "The Influence of Temperature and Radiation on Enzymic Reactions," "Chemical Statics in Enzyme Reactions," "Enzymic Syntheses," "Specificity of Enzyme Action," and an Appendix containing a very brief description of "Practical Methods," bring the volume to a close. There are both Subject and Author Indexes, but they are poor and ill-arranged, the irritating method having been adopted of following a name or a subject by a string of page numbers, without further differentiation or subdivision.

It is impossible for the reviewer to attempt the appraisement in detail of a book covering so large a field of work, but so far as he is able to judge from those portions dealing with branches of the subject with which he is familiar, much care has been taken in bringing the information well up to date, and in making a judicious selection from the original published work.

The author acknowledges his indebtedness to Mr. T. H. Pope, not only for his excellent translation, on which he is to be congratulated, but also for improvements and additions in the section dealing with practical methods, and for the references to the literature. H. F. E. HULTON.

ALLEN'S COMMERCIAL ORGANIC ANALYSIS, VOL. 5. A reply by W. P. DREPER to certain criticisms made by the reviewer (ANALYST, 1912, **37**, 385) appears in the *Journal of the Society of Dyers and Colourists*, vol. xxix., pp. 19-20.

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INSTITUTE OF CHEMISTRY.

PASS LIST: DECEMBER–JANUARY EXAMINATIONS.

Of twenty-seven candidates who presented themselves for the Intermediate Examinations held in Glasgow in December, and in London in January, eight passed: A. L. R. Clarke, B.Sc. (Lond.), G. N. Grinling, C. W. McHugo, H. A. Phillips, H. V. Potter, A. B. Stitch, B.Sc. (Glas.), H. E. L. Thomas and F. D. White. Of nineteen condidates who presented themselves for the Final Examination, fifteen passed. In the Branch of Mineral Chemistry: C. D. Barber, B.Sc. (Lond.), T. T. F. Cartwright, B.Sc. (Lond.), A.R.C.S. (Lond.), R. M. Doidge, B.Sc. (Lond.), F. M. Potter, B.Sc. (Lond.), A.R.C.S. (Lond.), and J. A. Watson, A.C.G.I.; in the Branch of Metallurgical Chemistry: F. G. Conyers; in the Branch of Organic Chemistry: K. C. G. Arbuthnot, B.A. (R.U.I.), J. C. Earl, and F. G. Tarn; in the Branch of the Chemistry of Food and Drugs, and of Water: J. W. Flint, B.A. (Cantab.), D. R. Frazer, H. E. Watts, Ph.D. (Zurich), B.Sc. (Lond.); for the Certificate: E. J. Wilson, M.A. (Cantab.), (F.I.C.); in General Chemistry: B. W. Drinkwater, A.R.S.M., A.R.C.S. (Lond.), and W. C. Ball, M.A. (Oxon.), Sc.D. (T.C.D.).

MARCH, 1913. Vol. XXXVIII., No. 444

THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

The annual general meeting of the Society was held on Wednesday evening, February 5, in the Chemical Society's Rooms, Burlington House. The President, Mr. L. Archbutt, F.I.C., occupied the chair.

The minutes of the previous annual general meeting were read and confirmed.

Messrs. N. Evers, B.Sc., and W. H. Simmons were appointed scrutators of the ballot papers for the election of officers and Council for 1913.

The Hon. Treasurer presented the accounts of the Society for the year 1912. These were adopted, and votes of thanks were passed to the Hon. Treasurer, Hon. Secretaries, and Auditors.

On the motion of the President, a vote of thanks was passed to the President and Council of the Chemical Society for their kindness in allowing the Society the use of their rooms at Burlington House during the past year.

The President delivered his Annual Address.

Mr. E. W. Voelcker moved that a hearty vote of thanks be accorded to the President for his address and for his services in the chair during the year, and that his permission be asked to print the address in the Analyst.

Dr. Monier-Williams seconded, and the motion was carried unanimously.

The President, having received the report of the scrutators, announced that the officers and Council for 1913 had been elected in accordance with the Council's nominations, the list being as follows:

President.—Leonard Archbutt, F.I.C.

Past-Presidents serving on the Council (limited by the Society's Articles of Association to eight in number).—Edward J. Bevan, F.I.C.; Bernard Dyer, D.Sc., F.I.C.; Thomas Fairley, F.I.C.; W. W. Fisher, M.A., F.I.C.; Otto Hehner, F.I.C.; R. R. Tatlock, F.I.C.; E. W. Voelcker, A.R.S.M., F.I.C.; J. Augustus Voelcker, M.A., B.Sc., Ph.D., F.I.C.

Vice-Presidents.—George Embrey, F.I.C.; J. T. Hewitt, M.A., D.Sc., Ph.D., F.R.S.; W. H. Willcox, M.D., B.Sc., M.R.C.P., F.I.C.

Hon. Treasurer.—Edward Hinks, B.Sc., F.I.C.

Hon. Secretaries.—A. Chaston Chapman, F.I.C.; P. A. Ellis Richards, F.I.C.

Other Members of Council.—R. M. Clark, B.Sc., F.I.C.; James Connah, B.Sc., F.I.C.; John Evans, F.I.C.; George T. Holloway, A.R.C.Sc., F.I.C.; G. D. Lander, D.Sc., F.I.C.; Thomas Macara, F.I.C.; G. W. Monier-Williams, M.A., Ph.D., F.I.C.; Cecil Revis; Harry Silvester, B.Sc., F.I.C.; F. Wallis Stoddart, F.I.C.; Arnold R. Tankard, F.I.C.; John White, F.I.C.

Messrs. R. G. Grimwood and Harley F. Knight were appointed auditors of the Society's accounts for 1913.

The ordinary monthly meeting followed the annual meeting, the President occupying the chair.

The minutes of the previous ordinary meeting were read and confirmed.

Certificates of proposal for election to membership in favour of Messrs. J. A. Goodson, F.I.C., F. W. Skevington, and J. C. White, A.I.C., were read for the second time; and certificates in favour of Messrs. Arthur Cecil Bescoby, M.A. (Cantab.), 24, Saumarez Street, Guernsey, Official Analyst to the States of Guernsey; Thomas Walter Frith Clark, 11, Quadrant Road, Canonbury, N., Chemist to Messrs. Barnett and Foster, Manufacturing Chemists; Frank Edward Day, B.Sc. (Lond.), A.I.C., Lansdowne, Limerick, Chemist to the Condensed Milk Company of Ireland; and James Fowler Tocher, B.Sc., F.I.C., 41½, Union Street, Aberdeen, Official Agricultural Analyst for the County of Aberdeen, Public Analyst for the Counties of Aberdeen and Banff.

Dr. George T. Beilby, F.R.S., F.I.C., was elected an honorary member of the Society; and Messrs. S. Elliott, A.I.C., T. R. Greenough, B.A., A.I.C., H. Lowe, M.Sc., A.I.C., J. P. Ogilvie, and J. A. L. Sutcliffe, A.I.C., were elected members.

The following papers were read: "Antipyrin in Toxicological Analysis," by G. D. Lander, D.Sc., F.I.C., and H. W. Winter; "A New Method for the Volumetric Estimation of Chromium, Vanadium, and Iron in Admixture," by Frederick W. Atack, M.Sc.; and a "Note on Hardened Oils," by Arthur W. Knapp, B.Sc., F.I.C.

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ANNUAL ADDRESS OF THE PRESIDENT.

(*Delivered at the Annual General Meeting, February* 5, 1913.)

WHEN I allowed myself to be nominated as your President, I am afraid I did not sufficiently realise the difficulty of a non-Public Analyst, and one who is not even in private practice, holding the Presidency of a Society which, in spite of the extension of its title, still is, and must always be, so closely indentified with the Public Analyst and his work. If, therefore, my remarks do not adequately deal with matters which are uppermost in the mind of the Public Analyst, I must crave the indulgence of that branch of our members. The field of analytical chemistry is so vast and varied that it becomes more and more difficult to keep in touch with all branches of it; one can, in fact, picture the Society in the future divided into sections, and its roll of membership immensely wider than it is at the present time.

I am pleased to report that during the past year we have elected nineteen new ordinary members, including one lady member, and your Council has reinstated one old member who elected to pay five annual subscriptions which were in arrear instead of being renominated, a tribute to the value of our journal. Three names have been removed from the roll owing to resignations and non-payment of subscriptions; and five, I regret to say, by death. Our total membership has, therefore, increased during the year from 397 to 411, and now includes thirteen honorary and 398 ordinary members.

As I have already hinted, I think we ought to have a much larger membership. I am afraid the amplification of our title which was made when the Society was incorporated in 1907 has not entirely removed the misconception that we are essentially a Society of Public Analysts. I hope, however, that the honour you have done me in electing me as your President will be understood, as it was doubtless intended, to be a recognition of the fact that our members include representatives of all branches of the analytical profession, and that we welcome to our ranks all qualified analytical chemists. That this was the intention of the founders of the Society can be in no possible doubt, for I find by reference to the first volume of our "Proceedings," dated 1876, the following words on page 6: "By the constitution of 'The Society of Public Analysts,' *all analytical chemists, but no others*, are eligible for membership" (the words in italics are so printed in the original). If all analytical chemists would join our Society and communicate their purely analytical papers to us, it would be one step towards that classification of chemical literature which is so desirable and will very soon become imperative. I regret to find that even our own members do not always bring their analytical papers here, and I commend to them the suggestion that they should make a point of doing so whenever possible, and should ask their friends to join us and do the same. I do not urge this from a selfish point of view, but in view of the convenience of having all analytical papers in one journal.

It is with great regret that I have to record the loss by death of five members: Arthur Edward Ekins, John Pattinson, Benjamin Edward Reina Newlands, Henry de Mosenthal, and Arthur Crozier Claudet. Mr. Ekins, who was elected a member in 1882, served twice on the Council and also as a vice-president, was one of those men of extraordinary energy who do not seem to allow themselves sufficient rest. He was a very regular attendant at our meetings, and personally I miss very much his genial presence. Mr. John Pattinson must, I think, have been an original member. He was one of the first Public Analysts appointed and a vice-president of our Society in 1896-97, but he was probably most widely known among chemists by his volumetric method for the estimation of manganese. Mr. Newlands was not a Public Analyst, but a technical chemist of great repute, who, though a member of our Society, was seldom seen at our meetings. The same remarks apply to Mr. de Mosenthal, who distinguished himself chiefly in connection with the chemistry of explosives. Mr. Claudet was one of our best known and most widely respected metallurgical chemists, assayer to the Bank of England and the Royal Mint. He took a great interest in various educational movements connected with metallurgical chemistry, to which he freely gave his time, and many of which he generously helped financially.

During the past session thirty-one papers were read at our seven meetings, of which thirty have been published in our journal; five other papers were communicated during the recess, making thirty-five published papers altogether, of which nine are on food and drugs and twenty-six on general subjects. Seven of the latter papers have contained the results of work carried on under the Society's Analytical Investigation Scheme; and as we do not present a medal or give prizes for this work, I feel sure I am interpreting your wishes in giving the titles of these papers, and thanking their authors from this chair for their work. They are:

"The Separation of Arsenic from Antimony and other Metals, with some Applications to Toxicological Work." By Stanley W. Collins, B.Sc., F.I.C.

"The Analysis of Lithopone." By W. L. Austin and Charles A. Keane, D.Sc., Ph.D., F.I.C.

"On the Effect of Lime on the Ammonium Molybdate Method of Lead Assay." By C. O. Bannister, A.R.S.M., and W. McNamara.

"Solubilities of the Lead Salts of the Higher Fatty Acids in Ether and in Petroleum Ether." By G. A. Neave, M.A., D.Sc.

"The Detection and Estimation of Arachis Oil." By Norman Evers, B.Sc., A.I.C.

"Alcoholysis and the Composition of Cocoanut Oil." By G. D. Elsdon, B.Sc., A.I.C.

"The Estimation of Citric Acid in the Presence of Certain Other Acids." By L. Gowing-Scopes.

In selecting these papers for special mention, my only desire is to encourage our younger members to offer their services for this very useful investigation work. We are, of course, equally indebted to all who contribute to our proceedings and assist in the progress of analytical chemistry. Mr. Chapman, who, as you know, originally suggested the investigation scheme, asks me to state that several investigations are still in hand, the results of which will be published in due course.

Among other papers read during the session, mention should be made of the valuable contribution to our knowledge of Chinese and Japanese wood-oils contained in the paper by Chapman. The differences in character between these two remarkable oils are clearly established by this paper, and we now have a much more exact knowledge than hitherto existed of their characteristic features and range of analytical values. I should like also to refer to the paper by Levy, published in our journal during the recess, in which the author shows that the slightly higher results obtained when the carbon in steel is determined by direct combustion than when the indirect method is used are due to the presence in the metal of dissolved carbonaceous gases. Although the point is more of academic interest than practical importance in the great majority of cases, it is satisfactory to have an explanation of the slightly different results obtained by the two methods; and Mr. Levy has done good service in calling attention to the matter, as the examples he gives show that in exceptional cases it may be of real importance.

A point of kindred nature, relating to the influence on analytical processes of gases occluded by or dissolved in metals, has been raised by Heath (*J. Ind. Eng. Chem.*, 1912, **4**, 402; ANALYST, 1912, **37**, 375), who shows that error may arise in the determination of oxygen in copper by direct combustion owing to the occlusion of hydrogen and other gases by the copper, and he has proposed a most elaborate method for obviating this error. Heath's method is so elaborate that it would be a very serious matter to have to use such a process in commercial work. In the paper which I read before the Society in 1900 there was no indication of any such error in the assay for oxygen of "tough-pitch" copper, since the loss of weight of such copper by simple heating to redness in hydrogen agreed very closely with the oxygen calculated from the weight of water found. As, however, the results obtained by analysts in the estimation of oxygen in copper have been called into serious question on several occasions recently, I think the subject is one which might be usefully investigated under our Analytical Investigation Scheme.

Our Hon. Treasurer is to be congratulated on the excellence of his report. It is very satisfactory to know that in spite of the depreciation of our securities, and increased expenditure, our financial position is better than last year by about £90, and that the increased expenditure includes a small salary for much-needed clerical assistance to our Hon. Secretaries. Mr. Richmond informs me that we now have about £200 available for investment from the income of the past three years, after providing £75 towards the cost of the next General Index, and we are much indebted to him for the very careful way in which he has husbanded our resources. In his successor, Mr. Hinks, we have a member who has the interests of the Society at heart, and will, I am sure, serve us well.

There is another matter connected with the accounts to which I wish to refer. The Research Fund, you will notice, shows a deficit—small, it is true, but if we had not had a considerable balance over from last year the deficit would have been much larger. Mr. Richmond thinks, and I agree with him, that we ought to try and raise a larger sum than £100 for this fund, as the interest on this is not sufficient to carry on the work, and it is not desirable that we should have to encroach upon the capital. I would therefore appeal to those members who appreciate the work that is being done, and realise the importance of it to the Society, to help us in raising a larger capital sum for this fund.

It is satisfactory to know that our journal has cost very little more than last year, and that the increased cost has been more than balanced by the increased revenue of the Society. The value of the journal has been fully maintained, every analytical paper of importance which has appeared in other journals having been abstracted. We are much indebted to our editor, Mr. Julian L. Baker, and the Publication Committee for the care and attention bestowed upon this, the principal asset of the Society. There is one point about the journal which particularly strikes me, and that is that the reviewers really do read the books sent to them for review. Whether one agrees or not with the trenchant criticisms of books which appear from time to time in the pages of the ANALYST, one does feel that they are at any rate honest attempts to tell what the reviewer believes to be the truth, and what he feels that the reader desires to know. The estimation in which the journal is held is shown by the fact that we have 153 subscribers outside the Society, besides about 35 copies which are sent as exchanges, and to institutions, etc.

The Chemical Society having invited us to send delegates to a Conference convened for the purpose of considering whether the present system of duplication of abstracts could be avoided, Mr. Hehner and I attended a meeting held in July last, at which certain preliminary inquiries were agreed upon. There is necessarily some difference of opinion as to what an abstract should give; some favour very brief abstracts, considering that those who wish for fuller information should consult the original papers; others feel that fuller abstracts are required for the benefit of those who have not the time and sometimes not even the opportunity of consulting the original. In either case the present system of duplication is wasteful, and it is devoutly to be hoped that the Conference may lead to some way of avoiding it.

I gather from Mr. Blount, to whom our thanks are due for representing us at the Eighth International Congress of Applied Chemistry held in New York in September last, that no analytical papers of striking importance were read. Those which appeared to be of sufficient merit have been secured by our editor for abstraction into the ANALYST.

I hope we shall have some good papers during the ensuing session, and I would appeal to all our members to ask themselves whether there is not some subject on which they could contribute to our proceedings. There is no lack of matter. To mention only a few problems awaiting solution in connection with my own practice, a reliable method for the direct estimation of rubber in vulcanised rubber goods is much needed. A good summary of our present knowledge in regard to this is contained in a recent paper by W. A. Ducca in the *Journal of Ind. and Eng. Chem.*, reprinted in the *Chemical News* of January 3, 1913. Notwithstanding the valuable work done by Henriques, Weber, and others, our present methods of analysis of manufactured rubber goods are cumbrous and far from satisfactory. One has only to peruse the paper by Beadle and Stevens in volume xxxv., page 11, of our journal to perceive the pitfalls which await the unwary. The advance must come from those who are in touch with manufacturing operations, and these are guarded with jealous secrecy. I cannot think, however, that manufacturers would lose by assisting in the elucidation of analytical problems. I hope before long we may have some further papers from those of our members who specialise in this difficult branch of analytical work.

Another direction in which progress is needed is in methods for the estimation of oxides and other non-metallic impurities in metals and alloys. Professor Turner has shown recently, in a paper read before the Institute of Metals, that there is no method for estimating oxygen in brass. Mr. E. F. Law has frequently called attention in papers read before the Metallurgical Societies to the neglect by analysts of the non-metallic impurities which are so plainly seen in micro-sections of metals and alloys, and which may have a quite important influence in obscure cases of corrosion, fracture, etc.

In the analysis of lubricating oils there is room for much improvement. In spite of the enormous extent to which gas and oil engines and super-heated steam engines are now used, and the consequent demand for oils which will lubricate at high temperatures without decomposing and forming serious deposits of carbon and hydrocarbons in the cylinders and valve chests, there is no recognised reliable laboratory method for ascertaining quantitatively what the behaviour of an oil will be under these exacting conditions. High viscosity does not seem to be so important for such oils as sufficient volatility for the oil to evaporate when it has done its work.

Viscosities of lubricating oils should be stated in absolute measure and in the form of a temperature curve, as recently urged by Messrs. Dunstan and Stevens, though the same thing was advocated by Mr. Deeley and myself twelve years ago, and a method described for calibrating commercial viscometers with mixtures of glycerol and water of known viscosity, so as to enable the viscosities of lubricating oils to be determined by means of such simple instruments. We published a table giving the viscosities in absolute measure of glycerol, water, and mixtures of the two in all proportions, which has never, so far as I am aware, been checked by other observers. It is desirable that this should be done, and that others should test our suggested method of determining absolute viscosities with commercial instruments, as Ostwald's viscometer is not a convenient instrument to use for commercial work. It is also important to bear in mind that unless the capillary tube of the viscometer sold as Ostwald's complies with certain necessary dimensions, the results obtained by it will not be correct.

So long as the present system of reporting "times of efflux" instead of "viscosities" is adhered to, the accurate calibration and standardisation of commercial viscometers is a pressing need. In this country the Redwood viscometer is the standard, and very large numbers of these instruments are in use, but unfortunately there is a lamentable want of agreement between their indications. I am glad to know that the National Physical Laboratory has this very difficult matter in hand at the present time, and I hope before long they will undertake the standardisation of these instruments. I may mention that this work is being done in connection with the International Commission for the Unification of Tests on Petroleum Products, and that a most interesting report on flash-point apparatus by Dr. Harker and Mr. Higgins was issued in 1911. Everyone who has to determine flashing-points of oils should study this report.

Among the subjects which have engaged the attention of the Council during the year has been the question of the fees paid for public analytical work and the tendency to reduce rather than increase the already inadequate remuneration offered for such work by public bodies. A glaring instance occurred in the early part of last year in connection with the appointments of Public Analysts for the Metropolitan Boroughs of Lambeth and Wandsworth and the County of Antrim. In the two former cases the fees offered amounted to 5s. per sample, the analyst being required to provide his own laboratory and equipment as well as a competent deputy during absence; and in the latter case the princely salary of £150 per annum was offered for the analysis of an indefinite number of samples—probably exceeding 800 per annum. It being obvious that the fees offered were insufficient to pay for the adequate performance of the work, the honorary secretaries were instructed to write to the two Borough Councils on the subject, and so serious did the matter appear that the Council of the Institute of Chemistry were led to move in the matter, and not only issued a public protest, but urged every Fellow of the Institute to refrain from applying for or accepting either of the three appointments. This led to the withdrawal of all the candidates except three for Lambeth and two for Wandsworth, of whom one was eventually appointed. Our letter of protest merely received a formal acknowledgment, and all the efforts of the Institute of Chemistry to obtain interviews with the Borough Councils and the Local Government Board failed, the latter being apparently satisfied with some private information which led them to regard the arrangements eventually made as satisfactory. In the case of Antrim an analyst was appointed, but subsequently resigned, as indeed he must do, unless he were prepared to give both his services and his private income for the benefit of the community. He has since been re-appointed on somewhat better terms.

The state of affairs thus disclosed is most unsatisfactory, quite as much so from the public point of view as from that of our profession. Men of the necessary high standing and qualification will not offer their services for inadequate pay, or if they do so, it is only because the appointments bring other work, and thus in the end private individuals have to pay for public work, which is quite wrong in principle. The acceptance of public appointments at inadequate fees tends to lower the analyst in the public estimation, and prejudices also the interest of the industrial chemist, who is already in many cases most inadequately paid. Matters seem to be far worse in Scotland than in England, and shockingly bad in Ireland. The Association of Public Analysts of Scotland, I am glad to say, have addressed a strongly worded and able statement of their case to the President of the Scotch Local Government Board, in which they allege that the low fees in that part of the country are not wholly unconnected with a desire on the part of certain members of the local authorities that the Adulteration Acts should not be worked, lest their trade should be interfered with. Whatever the reason may be, the persistent opposition to the working of the Acts in Scotland disclosed by the Scotch circular is a grave scandal, and the Local Government Board will fail in its duty to the public if full inquiry is not made into the facts and every support given to the analysts if their complaints are found to be justified.

One reason why analysts are so badly paid arises, no doubt, from the general ignorance which prevails in regard to our work, even in quarters where one would expect a more intelligent appreciation of it. How frequently it happens that the salary offered to a chemist entering a works is so meagre as to suggest that the main object is to pay the chemist as little as possible rather than to secure the services of a really capable man! Public Analysts rightly object to any interference with their work on the part of Medical Officers of Health, and to any encouragement being given to this by the Local Government Board, as contrary to the intention of the Act; but it frequently happens in industrial concerns that chemists holding somewhat similar, though not official, positions are subordinated to other officers, instead of being placed upon a footing of equality with them in relation to the board of management. It is, in my opinion, the duty of every chemist, official or otherwise, to take every opportunity of claiming the same recognition for his profession as is accorded to the professions of medicine, the law, and engineering. It is of great importance to the well-being of the community that the status as well as the remuneration of chemists should be such as to attract to the profession men of the requisite ability, standing, and integrity.

The time having come when, in the opinion of the Council, an attempt might be made to formulate a statement indicating the minimum fee or fees which should be accepted by analysts applying for appointments under the Sale of Food and Drugs Acts, a committee was appointed to consider the matter, and inquiries were addressed to all Public Analysts in the United Kingdom asking for information as to the number of samples analysed by them per annum, and the fees and other remunerations received in respect thereof. The Council wish to thank the large number of analysts who replied to these inquiries. The conclusions of the committee, arrived at after careful consideration of the whole subject, have been adopted by the Council; but as the question is merged in one of wider scope which is being considered by the Council of the Institute of Chemistry as a result of the Conference which was held in June last, it was decided to send a copy of your Council's report to the Registrar of the Institute, and to await the result of the deliberations of that body before making the conclusions public.

A matter of considerable importance has been called attention to by Dr. Dunn in a letter to the Hon. Secretaries, to which it is the desire of the Council that I should refer. In the terms of appointment of the Public Analyst for Lambeth and Wandsworth a distinction was made between "formal" and "informal" samples, and one of the candidates is reported to have remarked that the unofficial (or informal) samples would not demand so much time or trouble as the official samples. It seems to me very unfortunate that the taking of unofficial samples should ever have been encouraged. The only difference between them and the official samples is that if adulteration is detected the percentage of adulterant does not need to be determined in the case of the informal sample; but just as much care is needed in the one case as in the other to detect the adulteration. Supposing, for example, the percentage of adulterated samples is 10, it follows that 90 per cent. of all the samples, formal and informal, would have to be put through precisely the same tests, and would occupy exactly the same amount of time and trouble. The impression that informal samples can be easily dealt with appears to be held even by the Local Government Board, who in one of their recent circulars refer to examination by "rough sorting methods." The fallacy that an analyst nowadays can do his duty to his employers, public or private, by the use of "rough sorting methods" should have been exploded long ago. The matter has been very well put by the *Lancet:* "No analyst nowadays, if he is an honest man, dare certify that a sample, say, of butter, is genuine without determining not only the amount of butter-fat present, but the nature of the fat also, a process which involves considerable skill and attention. Then he will estimate the amount of water, curd, salt, and will search for preservatives, of which there are so many, and when found he will be bound to estimate the quantity in order to be able to say whether that is permissible. . . . The days when 10s. 6d. was an ample reward for casting a specimen of suspect coffee upon the surface of water and noting whether some of the particles rapidly sank, colouring the water on their downward way, which means the presence of chicory, are gone. The Public Analyst's work now is work of the highest scientific order; it must be done by a competent and conscientious man, otherwise the administration of the food laws must soon fall into disrepute. Slovenly practice must be impossible if discredit of the public analytical service is to be prevented. And there should be no temptation, in the shape of totally inadequate pay, to shirk the carrying out of a duty in a thorough and effective manner." I commend these words to our public authorities, Government and other. Scientific adulteration is most successful in eluding detection by "rough sorting methods," and the fees which are adequate to pay only for the use of such methods might as well be saved altogether.

In concluding this address, I wish to sincerely thank our Hon. Secretaries, and especially Mr. Chapman, for the kind way in which they have helped me to fill this chair during the past year. One needs to pass through the presidential chair to fully realise what the honorary officers do for the Society, and how deeply we are indebted to them for the voluntary time and labour which they so freely give.

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ANNIVERSARY DINNER.

THE Anniversary Dinner of the Society took place on Tuesday evening, February 4, at the Trocadero Restaurant, under the chairmanship of the President, Mr. L. Archbutt. There were present about eighty members and guests, among the latter being Professor R. Meldola, D.Sc., F.R.S. (President of the Institute of Chemistry), Dr. Bramley Taylor (Master of the Society of Apothecaries), Dr. R. T. Glazebrook, C.B., F.R.S. (Director of the National Physical Laboratory and President of the Faraday Society), Dr. A. W. J. MacFadden (Chief Inspector of Foods, Local Government Board), Dr. Frank Clowes (Chemist to the London County Council), Dr. R. Messel, F.R.S. (Acting President of the Society of Chemical Industry), Dr. Alexander Scott, F.R.S. (Treasurer of the Chemical Society), Professor A. W. Crossley, D.Sc., F.R.S. (Hon. Secretary of the Chemical Society), Professor A. K. Huntington (Acting President of the Institute of Metals), Professor H. G. Greenish, Dr. Arthur Voelcker, F.R.C.P., Mr. R. B. Pilcher (Registrar of the Institute of Chemistry), Mr. F. W. Beck, and Mr. Aubrey W. Rake.

After the loyal toasts had been duly honoured, the following toasts were proposed: "Government Departments," by Sir William A. Tilden, D.Sc., F.R.S., responded to by Dr. MacFadden; "The Society of Public Analysts and Other Analytical Chemists," by Professor Meldola, responded to by the President; and "The Guests," by Sir William Ramsay, K.C.B., F.R.S., responded to by Dr. Messel and Dr. Glazebrook.

ANTIPYRIN IN TOXICOLOGICAL ANALYSIS.

BY G. D. LANDER, D.Sc., F.I.C., AND H. W. WINTER.

INVESTIGATION CARRIED OUT UNDER THE SOCIETY'S ANALYTICAL INVESTIGATION SCHEME.

(*Read at the Meeting, February* 5, 1913.)

SUCCESSFUL toxicological analysis depends very greatly on the existence of good characteristic tests. The absence of such tests proves a most serious difficulty in the cases of veronal and sulphonal and its allies. Fortunately an excellent characteristic test for antipyrin is afforded by F. A. Steensma's reagent (*Pharm. Zeit.*, 1907, **52**, 851; ANALYST, 1908, **33**, 114), which is prepared by dissolving 1 grm. of *p*-dimethylaminobenzaldehyde in 100 c.c. of a solution of 5 c.c. of 25 per cent. hydrochloric acid in 100 c.c. of absolute alcohol. When a liquid containing a trace of antipyrin is evaporated to dryness, together with 5 c.c. of this reagent, a beautiful rose-red colour is developed. The colour is fairly permanent; it is dissipated by warming with sulphuric acid, but reappears on dilution. No reaction is given by commonly occurring substances, such as methyl alcohol, acetone, acetic acid, light petroleum, aniline, ethyl acetate, aldehyde, or formalin. Commercial amyl alcohol, however, yields a faint pink colour, as also does undenatured spirit, and this fact ought to be borne in mind when testing for antipyrin.

Extraction of Antipyrin.—The best solvent for extracting antipyrin is chloroform. A solution of 10 mgrms. antipyrin in 20 c.c. of water yielded, on extraction with 15 c.c. of chloroform, 8 mgrms. from the neutral or ammoniacal solution, and 5 mgrms. from the solution acidified by hydrochloric acid.

Similar experiments showed that light petroleum does not extract antipyrin, and that ether, ethyl acetate, and benzene, do so only in small proportions as compared with chloroform.

Extraction from Urine.—The following experiments illustrate the nature of the results obtained on extraction from urine without submitting the liquid to any process of purification, such as by means of basic lead acetate.

Five mgrms. of antipyrin were dissolved in 50 c.c. of human urine, rendered acid with tartaric acid, and extracted by the solvent mentioned in the tables.

ACID SOLUTION, 50 C.C.

Solvent.	Nature of Residue.	Reaction with Steensma's Reagent.
Light petroleum, 25 c.c. ...	nil	nil
Ether, 25 c.c.	dirty red	yellow
Ethyl acetate, 25 c.c. ...	dirty brown	yellow
Chloroform, 15 c.c. ...	fairly clean	yellow, red at edge

AMMONIACAL SOLUTION, 50 C.C.

Solvent.	Nature of Residue.	Reaction with Steensma's Reagent.
Light petroleum, 25 c.c. ...	colourless	nil
Ether, 25 c.c.	nearly colourless, crystalline	yellow, red at edge
Ethyl acetate, 25 c.c. ...	rather dirty, crystalline	yellow, red at edge
Chloroform, 15 c.c. ...	slightly green	distinctly red

Parallel experiments with normal urine, carried out in precisely the same way, showed that the nature of the various residues is very similar to that indicated above. On testing with the reagent, the colours obtained vary from yellow to dark brown when hot, fading in most cases to yellow when cold. The ether and ethyl acetate residues from the ammoniacal extraction sometimes give a pale red colour at the edges, which can be distinguished from the antipyrin coloration which forms in the liquid during evaporation. In a case of doubt it is advisable to make a parallel test with a portion of the residue to which a trace of antipyrin has been added.

Extraction from Viscera.—Four mgrms. of antipyrin were added to 4 ounces of putrid intestine of a dog and extracted with 300 c.c. of pure spirit and 3 grms. of tartaric acid for twelve hours in the cold. After removal of the alcohol in a vacuum the residue was taken up with 20 c.c. of water, filtered, and extracted twice with 20 c.c. of ether. The solution was then made ammoniacal and extracted with 20 c.c. of ethyl acetate, and then with 20 c.c. of benzene.

The combined residues from ether were very dirty, but gave the red coloration distinctly. The residues from ethyl acetate and benzene were in both cases fairly clean, and gave a good positive colour reaction.

This experiment shows that in the proportion given no difficulty need be experienced as regards the recovery of unaltered antipyrin in a routine Stas-Otto separation.

We then made a similar experiment, adding 1 mgrm. of antipyrin to 4 ounces of fresh intestine. The final solution was, however, extracted successively with light petroleum, ether, ethyl acetate, and chloroform, both from acid and ammoniacal solution. Ether and ethyl acetate remove most of the dirt, and the residues from chloroform extraction each gave a positive antipyrin test, that from ammoniacal being more intense than that from acid extraction.

The observations were repeated with 0·2 mgrm. of antipyrin to 4 ounces of intestine. In this case the results were negative.

Our observations show that Steensma's reagent serves to recognise antipyrin extracted from viscera according to the Stas-Otto process with certainty when as little as 1 mgrm. in 4 ounces is present; below this amount the results are doubtful or negative.

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A NEW METHOD FOR THE VOLUMETRIC ESTIMATION OF CHROMIUM, VANADIUM, AND IRON, IN PRESENCE OF ONE ANOTHER.

BY FREDERICK WILLIAM ATACK, M.Sc.Tech., B.Sc., A.I.C.

(*Read at the Meeting, February* 5, 1913.)

ATTEMPTS have been made to apply to the estimation of chromium and vanadium the method of reducing with zinc and hydrochloric acid, subsequently titrating with standard methylene blue solution, as used by Knecht and Atack for the estimation of molybdenum (ANALYST, 1911, **36**, 98), and by Atack for the estimation of tin in presence of antimony (*J. Soc. Dyers and Col.*, 1913, **29**, 9). It was not found possible to obtain a process for the estimation of vanadium based on the oxidation of VCl_2 to VCl_4 by methylene blue, owing to the slow rate at which the VCl_3 first formed is oxidised to VCl_4; indeed, the VCl_3 stage could be roughly distinguished by the change from rapid to slow disappearance of the methylene blue colour. The results obtained made it evident that the state of oxidation represented by V_2O_4 is stable in presence of methylene white, and the method now suggested is based on the oxidation of the leuco-dyestuff (methylene white) by means of vanadate (V_2O_5), resulting in the formation of methylene blue and V_2O_4. The amount of dyestuff so formed is then estimated, and corresponds to the oxygen available in the following equation:

$$2V_2O_5 = 2V_2O_4 + O_2.$$

Similarly, chromium cannot be estimated by reduction to chromous chloride, owing to the unstable character of this compound even in presence of a large amount of hydrochloric acid, but it has been found possible to use quantitatively the reduction of chromates to chromic salts on adding to methylene white:

$$4CrO_3 = 2Cr_2O_3 + 3O_2.$$

This method of oxidising a leuco-compound and estimating the dye produced is also available for the estimation of ferric salts, which are quantitatively reduced by methylene white to ferrous salts, and it has been found possible to combine the processes into a method for the estimation of chromium, vanadium, and iron, in presence of each other. In estimations in which leuco-methylene blue is used, nitric acid must not be present in the solution.

A convenient strength for the standard dyestuff solution is 4 grms. of fairly pure methylene blue (hydrochloride free from zinc) per litre. This approximately $\frac{N}{50}$ solution is readily standardised by one of the methods described elsewhere by the present author (*loc. cit.*), according to the reagents available. The method in which pure potassium chlorate is employed as the standard is to be recommended, a process identical with that described below for the estimation of chromate. A known volume of the standard methylene blue solution is heated to boiling with dilute hydrochloric acid in a current of carbon dioxide, and is then titrated whilst warm with titanous chloride solution until colourless. By this titration the titanous chloride solution is standardised, and the methylene white solution in the flask is ready for the addition of the oxidising agent to be estimated, standardisation and estimation being thus effected in the same flask. It is essential that an excess of methylene white be present to prevent the formation of chlorine and the consequent destruction of some of the methylene blue. Moreover, all methylene blue titrations must be carried out in an atmosphere of carbon dioxide, and in warm solution (over 40° C.) in order to obtain a sharp end-point. On account of the high tinctorial power of the methylene blue, the end-point is quite sharp, even if $\frac{N}{500}$ solution of the dyestuff and of titanous chloride are employed.

Estimation of Chromium.—The chromium salt is converted into chromate by oxidising with sodium peroxide, boiling to destroy the excess of the sodium peroxide, and then adding an aliquot portion of the chromate solution so obtained to a solution of methylene white prepared as above. The methylene blue so formed is estimated by titration with titanous chloride. The method has been tested on the purest Kahlbaum potassium dichromate, of which 1·3674 grms. were taken, dissolved in water, reduced with sulphur dioxide in presence of sulphuric acid, and made up to a litre after oxidising with sodium peroxide, the excess of which was destroyed by boiling. Fifty c.c. of standard methylene blue (1 c.c. = 0·00018 grm. oxygen) required 69 c.c. of titanous chloride, and on addition of 25 c.c. of the chromate solution further required 42·15 c.c. of titanous chloride for the methylene blue so formed. Addition of a further 25 c.c. of the chromate solution produced methylene blue equivalent to 42·2 c.c. of titanous chloride. One hundred and four c.c. of chromium are equivalent to 48 c.c. of oxygen. Thus, according to this method the sample contained 99·9 per cent. $K_2Cr_2O_7$.

Chromium in Presence of Iron.—Solutions containing iron and chromium are oxidised with sodium peroxide, boiled to destroy the excess of this reagent, and the precipitate dissolved by addition of dilute sulphuric acid. The solution is then made up to a known volume, and an aliquot portion added to methylene white solution, titration with titanous chloride then giving iron as ferric salt plus chromium as chromate. The iron is estimated by boiling a known volume of the solution with concentrated hydrochloric acid until the chromate has been completely reduced, making up to a known volume, and adding to methylene white. The method gave very satisfactory results with mixtures of standard iron alum and potassium dichromate. (See complete table later.) The method is available for the analysis of ferrochrome and of chromium steel. The powdered ferrochrome or chromium steel in the form of fine drillings are fused with caustic soda and sodium peroxide, and the melt extracted with water. The ferric hydroxide may be filtered off and the chromate estimated in the filtrate by addition to methylene white, or the iron and chromium may both be estimated by the process described above, in which case the chromium is determined by difference. The results of analyses of ferrochrome obtained by these processes agreed to within 0·2 to 0·3 per cent. on the chromium found.

Estimation of Vanadium.—The process employed is identical with that described above for chromium, oxidation being effected by sodium peroxide. The method has been tested on a pure (Kahlbaum I) ammonium metavanadate, NH_4VO_3, which should contain 77·8 per cent. V_2O_5. The sample was found to contain 77·6 per cent. V_2O_5.

Vanadium in Presence of Iron.—The process employed is similar to that used for the estimation of chromium in the presence of iron, the vanadium being estimated by difference. The method was tested on mixtures of known volumes of standard iron alum and ammonium vanadate solutions, and gave satisfactory results. (See complete table later.)

Chromium, Vanadium, and Iron.—It has been found possible to apply the above methods to the estimation of chromium, vanadium, and iron, in solution. The solution is oxidised with sodium peroxide, boiled, and made up to a known volume after addition of dilute sulphuric acid. (1) A suitable volume of the solution is added to methylene white, the methylene blue formed being titrated with titanous chloride. This gives the amount of chromate, vanadate, and ferric salt present. (2) Another portion is evaporated with concentrated hydrochloric acid to reduce the chromate and vanadate, the ferric salt remaining being estimated by addition to methylene white solution. (3) Finally vanadium, together with ferric salt, are estimated by reduction of a fresh portion of the solution with sulphur dioxide, boiling for a few moments, and then further replacing the sulphur dioxide by passage of a current of carbon dioxide through the liquid for a short time. There is no necessity to attempt a complete removal of the sulphur dioxide in this manner, as the solution is next oxidised with permanganate until it acquires a pink tinge, made up to a known volume, and the vanadate *plus* ferric salt estimated by addition of an aliquot portion to methylene white. The following results were obtained, using standard iron alum, ammonium vanadate, and potassium bichromate solutions :

	Found. Grm.	Present. Grm.
Vanadium	0·01750	0·01752
Chromium	0·00606	0·00604
Iron	0·01486	0·01483
Vanadium	0·01747	0·01752
Chromium	0·02413	0·02414
Iron	0·00745	0·00742
Vanadium	0·01776	0·01773
Chromium	0·01045	0·01050
Iron	0·01856	0·01850

In the analysis of chromium vanadium steel by these methods, the iron must be removed as hydroxide and estimated after solution in hydrochloric acid. It has been found possible to obtain a complete separation of the iron by fusing fine drillings of the steel with caustic soda and sodium peroxide. The melt is extracted with water, the whole being digested on a water-bath for some time, and then filtered through hardened filter-paper, finally washing with hot water. The filtrate contains the whole of the vanadium and chromium in the condition of vanadate and chromate, which are estimated by addition to methylene white of a portion of the solution. Another portion of the solution is acidified with sulphuric acid, filtered to remove silica if precipitated, reduced with sulphur dioxide, and then boiled for a short time. A current of carbon dioxide is passed through the solution, which is then oxidised with permanganate solution. A portion of this oxidised solution is added to methylene white to estimate the vanadium present. Fairly concordant results were obtained in three estimations of chromium and vanadium in a sample of steel:

	1.	2.	3.
Vanadium, per cent.	0·538	0·540	0·537
Chromium, per cent.	1·17	1·18	1·18

The presence of tungsten or molybdenum would vitiate the result.

Methods in which methylene blue is used have the advantage that this reagent may be used quantitatively in presence of hydrochloric as well as sulphuric acid. Moreover, owing to the excellent character of the end-point with titanous chloride in warm solutions, very small quantities of substance may be rapidly and accurately estimated by suitable adjustment of the strength of the titanous choride and methylene blue solutions used.

In conclusion, the author desires to thank Professor Knecht for the facilities placed at his disposal in carrying out this work.

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NOTE ON "HARDENED" OILS.

By ARTHUR W. KNAPP, B.Sc., F.I.C.

(*Read at the Meeting, February* 5, 1913.)

It is possibly a convenient time for calling the attention of analysts to the fact that in the immediate future they will be called upon to analyse certain new artificial fats, and, not improbably, to detect their presence as adulterants. For many years attempts have been made to convert into stearic acid the oleic acid which is produced in such large quantities as a by-product in the manufacture of "stearine" candles. The chemical reaction appears to be a simple one—the mere addition of hydrogen. This has at last been accomplished by making use of the catalytic action of platinum, palladium, or nickel. The same reaction which converts oleic acid into stearic acid also converts triolein into tristearin. Thus, for example, starting with olive oil, as the absorption of hydrogen proceeds, a turbid oil, then a liquid mess, then a soft fat, and finally a hard fat, is obtained. A similar change occurs with all oils containing glycerides of unsaturated acids. This rise in the melting-point is naturally accompanied by a decrease in the iodine value and refractive index. I obtained, amongst others, the following results with cottonseed oil, hardened with the help of a nickel catalyst (see p. 103).

Fats have been prepared in this way from cottonseed oil with iodine values as low as 5, and if desired the iodine value could doubtless be reduced to 0, and the melting-point raised to 60° to 70° C. Whilst it is too costly for commercial purposes to carry the saturation of the unsaturated glycerides to completion, it might be of value in the laboratory as an aid to determining the component glycerides in a pure oil. Not only the oils containing glycerides of oleic acid can be hardened, but also those containing glycerides of linolic acid and linoleic acid (the drying oils), and even of such highly reduced acids as clupanodonic (in whale oil). Anyone who has seen a malodorous oil converted into a bland odourless tallow realises the commercial possibilities of the process. And when it is remembered that the process can be stopped when the iodine value reaches a desired number, the possibility becomes evident of the preparation of a fat with any required analytical figures.

	Original Oil.	Hardened Oils.		
Appearance	Clear liquid	Solid particles, floating in oil	Soft greasy solid	Brittle solid
Butyro-refractometer (corrected to 40° C.)	57·7			47·7
Fatty acids				
Iodine value	110	94	55	22
Titer	34·7° C.	37·0° C.	42·5° C.	57·2° C.
Neutralisation value (mgrms. KOH)	197	196	196	192

The analyst is chiefly interested in the question of how these new fats are to be detected. It is doubtful if their most characteristic feature, the relatively high percentage of stearic glycerides which they contain, will be of much service. Until the manufacturer accomplishes the difficult process of completely removing the nickel, the detection of traces of this metal will be the simplest and most reliable test for hardened oils. Although the catalyst is very finely divided, the manufacturer can obtain a perfectly clear fat by careful filtration, and hence it is the nickel contained in the nickel soaps formed by the free fatty acids present that one has to detect. The following method is suggested: 50 grms. of the fat is heated in a flask with 20 c.c. hydrochloric acid, with continued vigorous shaking. The mixture is allowed to separate whilst hot, and part of the acid solution is evaporated to dryness, dissolved in a drop of water, and placed on a white tile. One drop of ammonium sulphide is added to this, and also to a drop of water for comparison. I have only tried this test on a few hardened oils, and in some cases with negative results. Dimethylglyoxime is a much more delicate test, but unfortunately Prall has found (Bömer, *Zeitsch. Untersuch. Nahr. Genussm.*, 1912, **24**, 104; and ANALYST, 1912, **37**, 452) that certain pure untreated oils give a red coloration. Hence further investigation is needed.

One of the most characteristic tests for fish oils—the bromide estimation—is quantitatively useless for these oils after hardening, as the percentage of ether-insoluble brominated glycerides is greatly reduced thereby. Not only are the analytical figures for the oils altered by this absorption of hydrogen, but also the traces of substances which often serve as a useful test for the particular oil in which they occur—*e.g.*, in Halphen's reaction (Bömer, *loc. cit.*). It is a remarkable and valuable observation that phytosterol and cholesterol are not changed in this process (Bömer, *loc. cit.*).

The three fats exhibited were obtained from a clear cottonseed oil. They were hardened by hydrogen with the help of different catalysts, and gave the following figures:

Catalyst.	Percentage of Catalyst in Oil.	Character of Product.	Butyro-Refraction (corrected to 40° C.).	Melting-Point: ° C.
Nickel	1·00	Hard	45·7	49
Platinum	0·10	Hard	47·8	46
Palladium ...	0·06	Brittle	45·5	52

The keeping properties of these hardened oils are remarkably good. The above have been prepared for nearly a year and a half, have often been exposed to damp air, and show no signs of rancidity. The free acidity (0·70 per cent. as oleic acid) has not appreciably changed.

Hardened oils give soaps which are good in colour, but lacking in lathering properties. It is unlikely that any objection will be raised to their admixture with other fats for soap-making. Their use in the preparation of edible fats is more open to question, and in the interests of public health certain investigations ought first to be made to show (1) that no harmful substances are produced by the chemical changes in the fatty glycerides and unsaponifiable matter; (2) that a high percentage of tristearin does not render the fat indigestible; (3) that traces of nickel, from 1 to 10 parts per million, are not harmful. There is perhaps little to be feared from objectionable substances in the hydrogen used, as the majority of such substances would poison the catalyst.

* * * * *

THE DETECTION OF ADULTERATION IN LINSEED OIL.

By G. D. ELSDON, B.Sc., A.I.C., and HERBERT HAWLEY, M.Sc., A.I.C.

Reply to Discussion. (See "Analyst," January, 1913, p. 3.)

We would point out that this method is not recommended so much as an additional test for some particular adulterant, but rather as a general test to which all linseed oils should conform, those oils not conforming being subsequently examined for the actual adulterant, oils passing the test being considered genuine without further examination.

The curve given in the figure is, as will be seen on reading the text, that for the maximum permissible extract obtained from linseed oils under the standard conditions given; the points for pure linseed oils will then, of course, all lie on or below this line.

The use of thinner paper might be an advantage, but we have found that continued extraction for many hours of the dried papers has given practically the same figure as that obtained by three hours' extraction.

The hexa-bromide method is at present being investigated by one of our colleagues.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Oil of Manketti Nuts. C. Grimme. (*Chem. Rev. Fett Ind.*, 1913, **20**, 1-3.)—Manketti nuts are the seeds of the fruit of *Ricinodendron Rautenii* (Schinz), a tree growing in various parts of German South-West Africa. The nuts examined contained 62·9 per cent. of kernels, which yielded 51·5 per cent. of oil, corresponding to 5·15 per cent. of the whole fruit. The oil was light yellow and viscous, and had a pleasant odour and taste. It gave the following analytical results:

	Oil.	Fatty Acids.
Sp. gr. at 15° C.	0·9286	—
Solidification-point	−8° to −10° C.	35° to 36° C.
Refractive index at 15° C.	1·4805	1·4694 at 50° C.
Acid value	0·94	—
Saponification value	194·80	—
Reichert-Meissl value	1·24	—
Polenske value	0·56	—
Iodine value (Wijs)	134·8	137·6
Insoluble fatty acids	94·8 %	—
Unsaponifiable matter	0·85 %	—
Glycerol	10·6	—
Melting-point	—	41° C.
Neutralisation value	—	196·5
Mean molecular weight	—	285·6

The oil had good drying properties, and would be suitable for the manufacture of varnishes, paints, etc. Physiological tests are required to ascertain whether it can be used for food purposes. C. A. M.

Estimation of Quinine by Titration with Picric Acid. E. Richter. (*Apoth. Zeit.*, 1912, 949-950, 960-961; through *Chem. Zentralbl.*, 1913, I., 194.)—The extraction of quinine from cinchona bark is more readily effected in an acid medium than by the use of alkali as prescribed in the German Pharmacopœia. The following method is shown to give reliable results: 3·75 grms. of the finely powdered bark are dried at 100° C., and heated for fifteen minutes in a 200 c.c. flask on the water-bath with 2·5 c.c. of 20 per cent. hydrochloric acid and 20 c.c. of water. When cold, the mixture is shaken with 25 grms. of chloroform and 60 grms. of ether, and after the addition of 10 c.c. of 10 per cent. sodium hydroxide the shaking is continued for fifteen minutes. An addition of 1 grm. of gum tragacanth is then made, and the shaking resumed until the layer of ether and chloroform becomes clear, after which 0·5 grm. of magnesium oxide is introduced, and 60 grms. of the solution (=2·5 grms. of bark) are filtered into a separating funnel. The clear filtrate is shaken twice with

20·2 c.c. and twice with 10 c.c. of $\frac{N}{10}$ hydrochloric acid, and the mixture of chloroform and ether then washed with three successive portions of 10 c.c. of water. The united acid extracts and washings are made up to 250 c.c., after evaporation of any dissolved ether on the water-bath, and 100 c.c. of the solution (=1 grm. of bark) shaken with 20 c.c. of water and 30 c.c. of $\frac{N}{20}$ picric acid solution until clear, after which the liquid is filtered and 50 c.c. of the filtrate titrated with $\frac{N}{10}$ potassium hydroxide solution, with phenolphthaleïn as indicator. Each c.c. of $\frac{N}{20}$ picric acid will precipitate 0·007725 grm. of quinine + cinchonine or 0·0081 grm. of quinine. If a represents the number of c.c. of $\frac{N}{10}$ alkali used in the titration of the 50 c.c. of filtrate, the percentage of alkaloid in the bark is obtained by the formula:

$$70 - 3a \times 0{\cdot}7725.$$

C. A. M.

Distilled Wool Fat. J. Marcusson and A. von Skopnik. (*Zeitsch. angew. Chem.*, 1912, **25**, 2577-2580.)—The products which are obtained when the fatty acids of wool fat are submitted to distillation with superheated steam at a temperature of 300° to 450° C. are known commercially under the names "wool-fat olein" (the liquid portion), "wool-fat ointment," and "wool-fat stearine." Wool-fat olein contains from 40 to 60 per cent. of unsaponifiable substances consisting of higher alcohols and unsaturated hydrocarbons; the fatty acids present are liquid. The wool-fat "ointment" consists of a mixture of solid and liquid fatty acids, together with from 16 to 33 per cent. of unsaponifiable substances. The solid fatty acids amount to about 50 per cent. of the total acids, have a melting-point of from 44° to 47° C., an iodine value of 9·9 to 15·1, and a molecular weight of 258 to 267; the liquid fatty acids have an iodine value of 43 to 48, and a molecular weight of 270 to 302. Wool-fat stearine contains from 32 to 42 per cent. of unsaponifiable substances, the remainder consisting of fatty acids having a melting-point of 58° to 67° C., an iodine value of 9·5 to 10·2, and a molecular weight of 318 to 382. W. P. S.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Estimation of Arsenic in Blood and Urine. F. Lehmann. (*Arch. Pharm.*, 1913, **251**, 1-4.)—A method of estimating arsenic described previously by the author and E. Rupp (ANALYST, 1912, **37**, 415) may be applied to blood and urine.

Blood.—Fifty grms. of the blood are treated in a Kjeldahl flask with 2·5 grms. of powdered potassium permanganate. After the lapse of ten minutes, 60 c.c. of concentrated sulphuric acid are added, and then 10 grms. of powdered potassium permanganate in small portions at a time. If the reaction becomes too violent, the flask is cooled under a stream of water. The contents of the flask are shaken for fifteen minutes, 30 c.c. of hydrogen peroxide solution are then added, and, after 7·5 grms. of anhydrous ferrous sulphate, 50 grms. of sodium chloride and 5 grms. of olive oil have been introduced, the mixture is distilled as described, the arsenic being collected in sodium bicarbonate solution and titrated with $\frac{N}{100}$ iodine solution.

Urine.—Five hundred c.c. of the urine are mixed with 2·5 grms. of potassium permanganate and evaporated to dryness. Frothing may be prevented by the addition of 0·5 grm. of paraffin wax. Five grms. of powdered permanganate and 20 c.c. of dilute sulphuric acid are mixed with the saline residue, and 20 c.c. of concentrated sulphuric acid are then added. When the evolution of gas has ceased, the mixture is heated with 30 c.c. of 3 per cent. hydrogen peroxide solution, and the arsenic is then distilled and titrated under the conditions given. W. P. S.

Estimation of Purine Bases in Blood. R. Bass and W. Wiechowski. (*Wien. med. Wochenschr.*, 1912, **25**, 1863-1864; through *Chem. Zentralbl.*, 1913, I., 331.)—The proposed method is based on the fact that uric acid and purine bases are not precipitated from very dilute solutions by means of phosphotungstic acid, provided that a minimum quantity of the latter is used for the precipitation of other substances present. The blood is treated with hirudin (an extract prepared from leeches' heads) to prevent it clotting, then diluted with three times its volume of water, and the mixture is heated to 80° C. on a water-bath, when partial coagulation takes place. After the addition of from 3 to 4 c.c. of 12 per cent. acetic acid for every 100 c.c. of blood taken, the heating is continued for a short time and the mixture is filtered. The filtrate is treated with 1 per cent. of its weight of concentrated hydrochloric acid, and small definite volumes of the liquid are mixed with varying amounts of 1 per cent. phosphotungstic acid solution in order to ascertain the quantity of this reagent which is necessary for precipitation without introducing an excess; the treated solutions are filtered, and the filtrates tested with albumin and 5 per cent. quinine hydrochloride solution. The test containing the largest amount of phosphotungstic acid without yielding a precipitate with the quinine-albumin reagent indicates the quantity of phosphotungstic acid to be added to the main volume of the blood-filtrate. When this has been done, the mixture is filtered, the filtrate is evaporated to 50 c.c., then rendered ammoniacal, and the phosphate is precipitated with magnesia mixture. The filtrate from the phosphate precipitate is treated with silver nitrate solution until a distinct precipitate of silver chloride is obtained; this is collected on a filter, washed with dilute ammoniacal silver nitrate solution, then decomposed with hydrogen sulphide, and the filtrate from the silver sulphide is acidified with hydrochloric acid and evaporated. Uric acid usually crystallises out, and may be collected and weighed; in cases where but little uric acid and a large quantity of purine bases are present, the two crystallise together, and the uric acid may be subsequently separated from the bases as ammonium urate.

Uric acid is a normal constituent of human blood; its quantity amounts to 1 to 2 mgrms. per 100 c.c. of blood, but varies considerably with different individuals. Ox blood also invariably contains uric acid, but this is not the case with horse blood. Purine bases are a constant constituent of human and animal blood, the larger quantity being found in human blood; the quantity of purine bases present is usually from four to ten times that of the uric acid. W. P. S.

Composition of Colostrum and of Colostrum Fat. A. Burr, F. M. Berberich, and A. Berg. (*Chem. Zeit.*, 1913, **37**, 69-71, 97-101.)—Analyses are recorded of colostrum obtained from twenty different cows, the results found being as follows: Sp. gr. at 15° C., 1·0330 to 1·0679; total solids, 11·83 to 31·11 per cent.; fat, 1·30 to

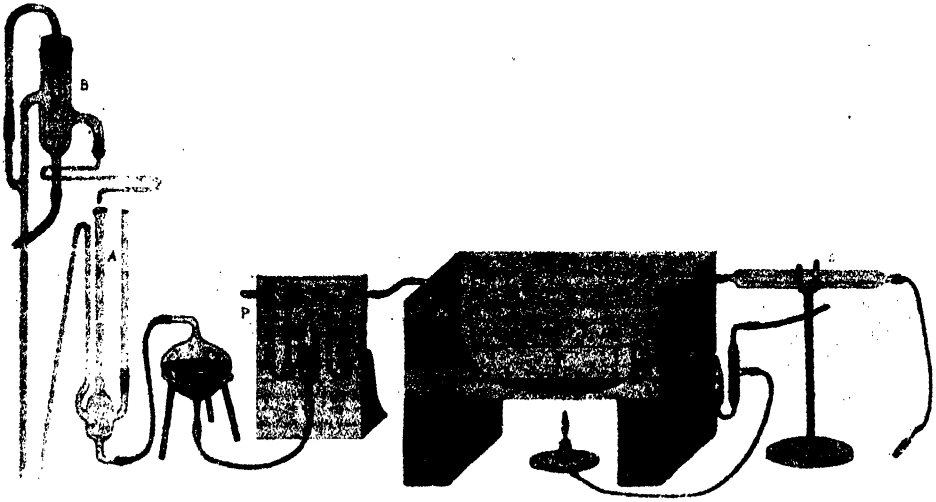
9·00 per cent.; solids-not-fat, 10·08 to 22·11 per cent.; total proteins (nitrogen × 6·25), 5·14 to 19·25 per cent.; casein (nitrogen × 6·37), 3·34 to 5·48 per cent.; ash, 0·82 to 1·37 per cent. In the case of nine of the samples the quantity of fat present amounted to less than 20 per cent. of the weight of the total solids, whilst in two cases the total solids contained 33·8 and 36·3 per cent. of fat, respectively. The Gerber method was found to be the best for the estimation of the fat; the Rose-Gottlieb method yielded low results, especially when the colostrum was rich in fat and total solids, and the Wollny refractometric method was unreliable. Results of analyses of the sera obtained from the samples of colostrum are also given; the sp. gr. of the sera varied from 1·0286 to 1·0727, and corresponded with the quantity of total solids present. The total solids ranged from 6·57 to 22·31 per cent.; the fat from 0·01 to 0·07 per cent.; and the ash from 0·87 to 1·31 per cent. As a rule the quantity of soluble nitrogenous compounds in the sera was slightly larger than in the corresponding colostrum.

The authors also separated the fats from the different samples of colostrum, and submitted them to examination. The fat appears to differ considerably in its composition from that of butter fat, the results obtained being—Refractometer reading at 40° C., 44·3 to 48·8; melting-point, 32° to 41° C.; Reichert-Meissl value, 11·5 to 29·1; Polenske value, 1·35 to 3·83; saponification value, 203·1 to 231·4; iodine value, 21·9 to 44·4. The fat contained 0·80 per cent. of cholesterol and 0·025 per cent. of lecithin. W. P. S.

Influence of Centrifugal Action on the Distribution of Cell Elements in Milk. R. S. Breed. (*Arch. Hyg.*, 1912, **75**, 383-392; through *Chem. Zentralbl.*, 1913. I, 321.)—Although it is possible to obtain apparently all the cell elements in the sediment when milk is subjected to centrifugal action in a machine rotating at 9,000 revolutions per minute, the separation of the cells is subject to the influence of other factors which cannot be clearly defined, and the number of cells found in the sediment bears no constant relation to the total number present in the milk. On this account, a correct estimate of the total number of cells in a sample of milk cannot be deduced from an examination of the centrifugal sediment, and methods in which the cells are estimated in this manner are of doubtful value. W. P. S.

Determination of Respiratory Quotients. L. Maquenne and E. Demoussy. (*Comptes rend.*, 1912, **155**, 881-886, 1055-1060.)—The determination of the real ratio of $\frac{CO_2}{O_2}$ in respiratory exchanges in exceedingly difficult, because the absorption and emission of the gases are not truly simultaneous. This is due to the different solubilities of oxygen and carbon dioxide, further complicated by a condition of supersaturation of the latter. To obtain correct results, it would be necessary either to extract before and after the experiment the whole of the dissolved and occluded gases, or else to leave the system at the end exactly in the same condition as at the start. Most experimenters have adopted the principle of confined air, which, although useful, can only give the apparent quotient, sometimes far removed from the real; and this difference will depend on the density of the charge—

i.e., the ratio of the volume of leaves or other tissue under investigation to the volume of the enclosed space. In such experiments it is obvious that the temperature must remain constant, as the solubility of the carbon dioxide is directly affected thereby. Two experimental principles are available for the determination of the apparent respiratory quotient, viz.: (1) By connecting two tubes together, one containing the leaves, and the other filled with mercury; it suffices then to reverse the system at the end of the period to obtain a sample of the gas surrounding the leaves; (2) by connecting the respiration tube by means of a stopcock with a smaller tube which has been completely evacuated, rapidly opening the cock, and thus taking an instantaneous sample of the gases. From an analysis of these gases the apparent quotient μ can be calculated, and if δ = the density of charge, and c the coefficient of absorption of the carbon dioxide, then the real quotient m is obtained from the formula $\mu = m \frac{1-\delta}{1-\delta+c\delta}$; by determining μ for two different values of δ it is possible to calculate the solubility c of carbon dioxide in the plant juice, which is approximately double the solubility in water at the same temperature. The accuracy of such a method depends on the rapidity of the gaseous exchanges, and the results are liable to be too low, particularly with thick, fleshy tissues. The authors now describe a new method, termed the "displacement method," which consists in passing a current of air through a tube charged with the leaves at a constant slow rate of speed, such that the gas issuing shall contain at least 2·5 to 3 per cent. of carbon dioxide. After a certain time, the juices becoming saturated with carbon dioxide, an equilibrium is established, and the composition of the issuing gases remains constant as long as the intensity of respiration and quotient $\frac{CO_2}{O_2}$ are constant; analysis then immediately yields the real respiratory quotient. The motive portion of the apparatus (see figure) consists of a sort of tantalus flask, A, fed by a fine jet of water issuing from



a capillary tube under absolutely constant pressure. The impulses derived from this apparatus are transmitted to a small mercury pump, P, which aspirates 2 to 4 c.c. of air at each oscillation, according to the adjustment, and pumps the air into the respiration tube T, containing the leaves, at the same time displacing the respiratory gases. The tube T is immersed in a water-bath at constant temperature, and the respiratory gases are collected by displacement in the tube C, care being taken to avoid variations in pressure; or else an instantaneous sample may be taken from the tube T itself in the manner indicated above. If it be assumed that the arriving air mixes immediately with the gases already present in the tube, the principle of the method will be readily understood. The time required for the establishment of the equilibrium depends on the capacity of the pump and the density of the charge; if the pump discharges a volume of air equal to the volume of the tube T in one hour, and the density of charge is 0·1, about four hours must elapse before a sample can be drawn for analysis. With organs which respire very actively, the action of the pump may be accelerated, and the operation completed in two hours; but with tissues respiring slowly, as with the leaves of fat-yielding plants, the current of air must be slowed down, so that the analysis cannot be made until after twenty-five to thirty hours. When the intensity of respiration remains constant for a sufficient length of time, this method gives results only very slightly lower than the real quotient; in the case of leaves in which the reserve material is very rapidly exhausted, the long duration of the experiment is a drawback and the quotient is too low. Comparing the displacement method with the vacuum method, it is noted that in the case of leaves with thin parenchyma, where gaseous exchanges take place easily and rapidly, either method may be used, since both give results in good concordance. In the case of fleshy leaves, stems, wood, or germinated seeds, where equilibrium with the outer air takes place with difficulty, the displacement method is the only one which affords a result closely approximating to the real quotient, the vacuum method yielding numbers which are much too low. J. F. B.

Estimation of Acetone in Urine. O. Sammet. (*Zeitsch. physiol. Chem.*, 1913, **83**, 212, 225.)—Results of a critical investigation of various methods for the estimation of acetone are recorded, particular attention being paid to the application of the methods to the estimation of acetone in urine. The first method examined was that proposed by Graaf (*Pharm. Weekblad*, 1907, **44**, 555), in which the urine is distilled and the acetone in the distillate precipitated by means of *p*-nitrophenylhydrazine in acetic acid solution; the precipitate is collected, dried at 105° C., and weighed. The precipitate has the formula $(CH_3)_2.C:N.NH.C_6H_4(NO_2)$, and 198 parts of it by weight correspond with 58 parts of acetone. This method was found to yield from 95·5 to 96·2 per cent. of the quantity of acetone actually present when the amount of acetone varied from 0·1 to 0·2 grm. per 100 c.c. of urine. Messinger's method also yielded satisfactory results (*cf.* ANALYST, 1908, **33**, 98). When this method is applied to urine, the latter should be treated with tartaric acid and distilled; calcium carbonate is then added to the distillate, and the mixture is again distilled. The second distillate is rendered alkaline with potassium hydroxide solution, an excess of $\frac{N}{10}$ iodine solution is added, and, after shaking, the mixture is acidified with hydrochloric acid and the excess of iodine titrated with thiosulphate solution. Each c.c. of $\frac{N}{10}$ iodine solution is equivalent to 0·000967 grm. of acetone. The yield obtained by the method was about 96 per cent. of the quantity of acetone present.

Oppenheimer has used a method, described originally by Denigès (*cf.* ANALYST, 1899, **24**, 92), for the estimation of acetone in urine; but the results obtained are about 10 per cent. too high when Oppenheimer's factor (0·055) is used for converting the weight of the precipitate into acetone; better results are obtained when the factor 0·0495 is employed. The volumetric method of estimating the mercury in the precipitate (ANALYST, 1896, **21**, 303) is untrustworthy.

The author has also carried out a number of experiments in which it was sought to estimate the amount of acetone from the volume of the precipitate obtained in Denigès's method; the precipitate was subjected to centrifugal action in a graduated tube until it no longer decreased in volume, and it was found that the total volume of the precipitate multiplied by 0·0912 gave the weight of acetone. This is only the case when the volume of the precipitate does not exceed 0·4 c.c., and even then the results are not strictly correct. W. P. S.

Comparison of the Farup and Schumacher-Jung Methods for the Estimation of Mercury in Urine. W. Beckers. (*Arch. Pharm.*, 1913, **251**, 4-7.)—Experiments carried out by the author show that Farup's method (in which the mercury is precipitated as metal by means of zinc-dust and subsequently with stannous chloride, then collected on a gilded asbestos filter, and weighed) and Schumacher-Jung's method (ANALYST, 1902, **27**, 368) yield practically identical results. W. P. S.

ORGANIC ANALYSIS.

Berlin (Prussian) Blue Reaction (Detection of Ferrocyanides and Cyanides). D. Vorländer. (*Ber.*, 1913, **46**, 181-192.)—It is shown that the reaction between ferric salts and ferrocyanide requires a considerable time for its completion, and that it is retarded by high acid or saline concentrations. When testing for ferrocyanide in very dilute solutions, ferrous sulphate is a far more sensitive reagent than ferric chloride. The pale colour of the ferrous solution allows the use of a large excess, which tends to carry the reaction to completion, and there is usually enough dissolved oxygen in the water to convert the ferrous ferrocyanide to ferric ferrocyanide, so that, with only traces of ferrocyanide present, the maximum intensity of colour is obtained instantaneously. G. C. J.

Estimation of Caoutchouc in Rubber Wares, particularly Insulated Wires. P. Goldberg. (*Chem. Zeit.*, 1913, **37**, 85-86.)—The direct estimation of caoutchouc in vulcanised rubber does not yield very satisfactory results, and, recognising this, the Verband Deutcher Elektrotechniker has adopted an indirect method for estimating the caoutchouc in wire insulations. The method approved is one adapted by Hinrichsen and Manasse, according to which 1 grm. of the material, after extraction with acetone and drying at 50° to 60° C., is boiled with 25 c.c. of petroleum (fraction 230° to 260° C.) until the caoutchouc is dissolved, after which the solution is diluted with benzene, and the insoluble matter is washed by decantation or centrifuging and finally weighed. This residue will include not only the mineral matters, but also any organic fillers, such as lampblack, cellulose, etc., and the sum of these together with the acetone extract (free sulphur and paraffin hydrocarbons) gives the total loading, which, deducted from 100, gives the vulcanised caoutchouc. From this, 3 per cent. is deducted for the combined sulphur in order to arrive at the pure caoutchouc. The objection to this method lies in the fact that caoutchouc-like bodies may remain undissolved in the residue, which may also be contaminated with charred products owing to the high temperature employed. In either case the percentage of true caoutchonc would be lowered, and differences up to 3 per cent. are often recorded. An estimation of the mineral loading matters by incineration in the ordinary way is scarcely possible, owing to the profound modification of many of the loadings used, under the action of the high temperature, the oxidising influence of the air, and the reducing influence of the carbon. Most of these inconveniences may, however, be avoided by performing the incineration in an atmosphere of nitrogen. A temperature of 400° C. is quite sufficient to decompose and drive off the caoutchouc in the form of volatile compounds, and under these conditions oxidation, reduction, separation of carbon or oxidation of a sulphide, need not be feared. The author describes the following procedure: 1 grm. of the material, extracted with acetone and dried at 50° to 60° C., is placed in a tared porcelain boat which is inserted in a glass tube 50 cm. long, open at both ends. One end is connected with a supply of dried nitrogen, and as soon as the air has been expelled a small flame is applied. The caoutchouc melts, decomposes, and distils, and the resulting vapours are drawn off by connecting the other end of the tube with a water air-pump. The operation is complete in about twenty minutes, and the boat is weighed, the caoutchouc being calculated as indicated above. Mixtures were made containing all the usual mineral loadings employed in vulcanised wares, and the estimations of caoutchouc in no case differed by more than 0·3 per cent. of the calculated percentages. Exceptions are—readily decomposable carbonates, such as magnesium carbonate, and red sulphides, such as golden antimony and vermilion. These cases must be dealt with specially: the carbonates by the insertion of potash bulbs to collect the carbon dioxide expelled; the golden antimony, which remains in the ash as the trisulphide, by an estimation of antimony in the residue and calculation as pentasulphide; and the vermilion, which volatilises as such, by a separate estimation. J. F. B.

Quantitative Separation of Mixtures of Certain Acid Coal-Tar Dyes. W. E. Mathewson. (*J. Ind. Eng. Chem.*, 1913, **5**, 26-29.)—The method proposed depends on the extraction of one of two dyes from an aqueous solution by means of an immiscible solvent, and the following procedure, or some simple modification of it, has been employed: The solution, containing from 0·2 to 0·4 grm. of the dyes, is treated with sufficient water and hydrochloric acid of known strength to bring its volume to about 50 c.c., and its acid concentration to that point at which the difference in percentage of colour extracted for the two dyes is near its maximum. The solution is then shaken out with the immiscible solvent, being passed successively through three or four separating funnels, each containing about 50 c.c. of the solvent. The portions of the solvent are washed with 50 c.c. of acid of the same concentration as the original solution, and this washing operation is twice repeated. The dye relatively more soluble in water is estimated in the combined washings and extracted solution. The second dye is removed from the solvent by shaking with water or very dilute sodium hydroxide solution, or, more quickly, with dilute sodium hydroxide solution, after the addition of a quantity of gasoline or similar substance in which the dye is insoluble. In many cases it will be found to be necessary to increase the number of successive extractions. In the following mixtures, each containing two dyes, from 97 to 102 per cent. of the quantities actually present were obtained; the solvent used and the acidity of the solution are also given: Naphthol Yellow S and Light Green S F Yellowish, solvent, amyl alcohol; acidity, 20 c.c. of 5 N. hydrochloric acid to 30 c.c. of solution. Ponceau 3 R and Naphthol Yellow S, solvent, amyl acetate; acidity, 20 c.c. of concentrated hydrochloric acid to 20 c.c. of solution. Amaranth and Orange I, solvent, amyl alcohol; acidity, 2 c.c. of 5 N. hydrochloric acid to 30 c.c. of solution. Light Green S F Yellowish and Ponceau 3 R, solvent, amyl alcohol; acidity, 20 c.c. of concentrated hydrochloric acid to 30 c.c. of solution. Ponceau 3 R and Indigo Carmine, solvent, amyl alcohol; acidity, 2 c.c. of 5 N. hydrochloric acid to 28 c.c. of solution. Light Green S F Yellowish and Indigo Carmine, solvent, dichlorhydrin; acidity, 2 c.c. of 5 N. hydrochloric acid to 23 c.c. of solution. Naphthol Yellow S and Amaranth, solvent, amyl acetate; acidity, 10 c.c. of concentrated hydrochloric acid to 10 c.c. of solution. W. P. S.

Comparative Investigation of Methods for the Estimation of Dyes. H. Salvaterra. (*Monatsh. für Chem.*, 1913, **34**, 255.)—Knecht's method (*J. Soc. Dyers and Colorists*, 1903, 169, and 1905, 111 and 292) consists in titrating the dye, which acts as its own indicator, with standard titanium chloride solution. In Pelet's method ("Die Theorie des Färbeprozesses," Dresden, 1910, p. 49 *et seq.*) a basic dye is precipitated with a standard solution of an acid one, the end-point being shown by the disappearance of one or both of the coloured rings produced by dotting on filter-paper. These two methods were applied to a number of carefully purified dyes. Both gave good results. The method of Knecht, which involves titration in an atmosphere of carbon dioxide, is less convenient to carry out than that of Pelet; but this, on the other hand, requires the provision of a large number of standard solutions. For dyed silk the Knecht method alone is capable of giving satisfactory results. Both methods are applicable to baths which have been used for dyeing, but the colorimetric method is quite useless for this purpose. O. E. M.

Quantitative Study of the Absorption of Ultra-Violet Rays by the Fatty Acids and their Isomeric Esters. J. Bielecki and V. Henri. (*Comptes rend.*, 1912, **155**, 1617-1620.)—In order to study the influence of molecular constitution on the absorption of ultra-violet rays by substances of the aliphatic series, the authors have investigated comparatively various isomeric bodies—*e.g.*, free fatty acids and esters having the same empirical formulæ, also various isomeric esters. Observations were made in aqueous and alcoholic solutions, the molecular absorption coefficients being tabulated for the various wave-lengths for each pair of isomerides examined. The results showed that the absorption spectrum in the case of the acids differs from that of the esters isomeric with them. This difference exists independently of the solvent; it is as distinct in aqueous as in alcoholic solutions. The absorption of the ultra-violet rays by acids and esters is not determined by the empirical formula. As the molecule becomes more complex by the addition of CH_2 groups, the absorption increases. The absorption depends on the actual constitution of the molecule, and is not only different for isomeric acids and esters, but also for esters isomeric with one another. J. F. B.

Fractional Precipitation of Fatty Acid Mixtures (Detection of Rape Oil). H. Kreis and E. Roth. (*Chem. Zeit.*, 1913, **37**, 58.)—The presence of rape oil, and possibly of cocoanut oil, in another oil may be detected by fractionally precipitating the mixed fatty acids of the oil with lead acetate and examining the fatty acids separated from the lead precipitate. A portion of the mixed fatty acids is dissolved in alcohol and treated with about one-tenth the quantity of lead acetate required for complete precipitation. The lead salts of the fatty acids thus precipitated are separated, decomposed with hydrochloric acid, and the melting-point, refractometer value, and molecular weight, of the liberated fatty acids are determined. The results given by the fatty acids obtained in this manner from various oils are shown in the following table:

Fatty Acids from—	Melting-Point: °C.	Refractometer Value at 60° C.	Molecular Weight.
Olive oil	About 53·0	17·0	
Earthnut oil	„ 56·8	19·5	
Sesame oil	„ 52·5	17·5	
Cottonseed oil	„ 58·4	15·0	
Rape oil	„ 30·0	32·0	About 340
Butter fat	„ 53·5	17·0	„ 273
Cocoanut oil	„ 37·5	11·0	„ 236
Tallow	„ 61·0	17·0	
Lard	„ 58·0	17·5	
(Erucic acid)	33–34° C.	—	338

The precipitate obtained in the case of rape oil fatty acids evidently consists principally of erucic acid, but the high molecular weight of the fatty acids indicates that a quantity of lignoceric acid is also present (*cf. J. Soc. Chem. Ind.*, 1898, **17**, 1009). W. P. S.

Detection of Histidine. K. Inouye. (*Zeitsch. physiol. Chem.*, 1913, **83**, 79-82.)—When an aqueous solution of histidine is treated with an excess of sodium carbonate, and diazobenzenesulphonic acid is added to the mixture, a cherry-red coloration is produced; 1 part of histidine in 100,000 parts of solution will give the coloration. The reaction may be employed for the detection of histidine when this is combined in a protein molecule, but the value of the reaction is influenced by the fact that tyrosine also gives a coloration under similar conditions. The interfering reaction of tyrosine may, however, be prevented by shaking the protein solution, after the addition of sodium carbonate, with a few drops of benzoyl chloride until the odour of the latter disappears, and then adding the diazobenzenesulphonic acid; a large excess of benzoyl chloride must be avoided or removed by continued shaking, or the histidine reaction will also be affected to some extent. Before testing a protein for the presence of histidine it must be hydrolysed, preferably by heating for six hours with hydrochloric acid; the hydrolysis may also be brought about by the action of trypsin, but in this case a control experiment must be made to ascertain whether the trypsin is free from histidine. The solution of the hydrolysed protein is evaporated to a syrupy consistence, dissolved in water, warmed after the addition of lead oxide, and then cooled. The mixture is next rendered alkaline with sodium carbonate, filtered, and a portion tested with the reagent. If a red coloration is obtained, another portion of the solution is shaken with benzoyl chloride, and again tested to insure that the reaction is due to histidine and not to tyrosine. Both histidine and tyrosine may be detected in Witte's peptone by means of the test; sturine yields only histidine and no tyrosine, whilst orzynine (a protamine obtained from the testes of the tunny-fish) contains only tyrosine. W. P. S.

Modification of the Official Method of Determining Humus. O. C. Smith. (*J. Ind. and Eng. Chem.*, 1913, **5**, 35-37.)—The official method states that 4 per cent. ammonia solution should be shaken with the soil for the first twenty-four hours, and then allowed to stand twelve hours and settle. The clearest part is then drawn off, filtered, and the determination made without further treatment. This filtering does no good, and it is rarely that sufficient clear solution is obtainable. It is better not to allow the sediment to settle, but to shake well and pour all the soil possible on to the filter, when, after about five or six hours as a rule, and sometimes less, a perfectly bright filtrate is obtainable. The first turbid runnings may be discarded or returned to the filter, a Schleicher and Schüll folded filter about 30 cm. in diameter, with hardened points, being found most suitable. Duplicate experiments usually agree within 0·1 per cent. H. F. E. H.

Behaviour of Nitroglycerine when Heated. W. O. Snelling and C. G. Storm. (*Technical Paper* 12, *Department of the Interior*, *Bureau of Mines*, *Washington.*)—The experiments were carried out in a heavy steel cabinet with a thick plate-glass window. Usually about 2 c.c. of nitroglycerine were heated in a small test-tube in a paraffin bath; the temperature was measured by means of a constantan-copper couple in the nitroglycerine. Results were as follows: Nitroglycerine begins to decompose at 50° to 60° C. At 70° C. commercial nitroglycerine reacts with potassium iodide starch paper in fifteen to thirty minutes. At 135° C. the nitroglycerine is coloured strongly red by oxides of nitrogen, produced by decomposition. The boiling-point varies one or two degrees with the sample, but is about 145° C. at normal pressure; ebullition is due partly to decomposition, and partly to volatilisation of the nitroglycerine. The heat produced by decomposition is sufficient rapidly to raise the temperature in the absence of cooling. At about 218° C., the temperature varying within about 5 degrees, according to the sample, explosion takes place. Nitroglycerine may be distilled at temperatures between 145° and 210° C.; the distillate consists of nitroglycerine, nitric acid, water, and other decomposition products, and the residue probably contains chiefly glycerol, with dinitroglycerine, mononitroglycerine, and other decomposition products. O. E. M.

Examination of Tung Oil. W. Hoepfner and H. Burmeister. (*Chem. Zeit.*, 1913, **37**, 18-19, 39.)—From the examination of a large number of samples, the authors conclude that the iodine value (Hübl) of commercial tung oil ranges from 156 to 171, the average being 164·6, whilst the average refractive index at 20° C. in Abbe's apparatus is 1·5175. These two values considered, in conjunction with the results of Bacon's test (Analyst, 1912, **37**, 546) give information as to the purity of a sample. In using Bacon's test it was found best to modify the time of heating to twelve minutes at 310° C. If under these conditions the oil becomes so solid that it can be powdered in a mortar, there can be no doubt about its being pure. On the other hand, a mixture containing as little as 5 per cent. of soya bean or teaseed oil remains viscid or fluid at the end of the heating. As a further test that the solid polymerised product is free from foreign oils, the fatty acids may be separated and their values determined (*cf.* Analyst, 1912, **37**, 410). C. A. M.

INORGANIC ANALYSIS.

Methods for the Preparation of Neutral Solutions of Ammonium Citrate. J. M. Bell and C. F. Cowell. (*J. Amer. Chem. Soc.*, 1913, **35**, 49-54.)—The method approved by the Association of Official Agricultural Chemists requires the use of an alcoholic solution of corallin as indicator, but this and purified litmus solution both give inaccurate results. The better method is that proposed by Hall and Bell (*J. Amer. Chem. Soc.*, 1911, **33**, 711), by which it is found that the solution when just neutral has the highest electrical conductivity. In the present paper two methods are described for the determination of the end-point in the titration of a weak acid by a weak base where the ordinary indicators fail, and both of them are simpler than that involving the conductivities of solutions which must be determined at constant temperatures. In the first method, an index of the excess of ammonia is obtained by shaking out with chloroform and titrating with a dilute acid. The distribution ratio of ammonia between chloroform and water is such that between equal volumes of water and chloroform, free ammonia will distribute itself about $\frac{1}{26}$ in the chloroform layer, and $\frac{25}{26}$ in the water layer. The proportion in which citric acid and ammonia solutions must be mixed to give a solution exactly neutral may be found by a graphic method in which there are plotted the number of c.c. of ammonia solution used against the number of c.c. of $\frac{N}{10}$ hydrochloric acid required to neutralise 50 c.c. of the chloroform extract. In the second method the rise in temperature due to the heat of neutralisation is observed as the titration proceeds, the end-point being at the break in the heating

curve. Experiments were carried out in a Dewar flask of about 200 c.c. capacity, provided with a platinum stirrer and a thermometer graduated in tenths of degrees. The exact neutral point is found by plotting a curve, using the number of c.c. of ammonia as abscissæ, and the rise of temperature as ordinates. The accuracy of the method was further confirmed by the direct titration of ammonia against $\frac{N}{2}$ acid.
H. E. E. H.

Separation of Arsenic and Tungsten. S. Hilpert and T. Dieckmann. (*Ber.*, 1913, **46**, 152-155.)—The authors have studied the various methods which have been proposed for this separation, and recommend the following: The solution (40 c.c.) is mixed with 15 grms. of cuprous chloride and 150 c.c. of concentrated hydrochloric acid (sp. gr. 1·16), and distilled as far as possible. A further 150 c.c. of acid is added and the distillation repeated. Potassium hydroxide (10 grms.) is added to the residue, and the mixture heated to dissolve the tungstic acid, on the surface of which 10 per cent. or more of the arsenic may be adsorbed. After cooling, 150 c.c. of acid is again added and a final distillation made. The mixed distillates are nearly neutralised with solid ammonium carbonate, and finally an excess of sodium bicarbonate is added and the arsenic titrated with iodine, or the arsenic in the distillate may be precipitated as sulphide and finally estimated as magnesium pyro-arsenate. Provided not more than 0·5 grm. of tungstic acid is present, the error of the above method does not exceed 0·5 mgrm. It is stated that larger amounts of tungstic acid seriously impair the accuracy of the results, but no figures are given in support.
G. C. J.

Estimation of Arsenic in Pyrites. E. Schürmann and W. Böttcher. (*Chem. Zeit.*, 1913, **37**, 49-51.)—A critical review of the methods which have been proposed for the determination of arsenic in pyrites. In the opinion of the authors, large errors, such as are occasionally recorded by critics of one or other of the methods in common use, can generally be traced to the process of opening up the ore. Whatever method be adopted for the final determination of the arsenic, they prefer to conduct the preliminary oxidation and solution as follows: About 3 grms. of the sample, which should be as finely divided as possible, is introduced into a 500-c.c. flask together with 12 grms. of crystallised chromic acid, and the mixture is covered with 30 c.c. of dilute (1 : 1) sulphuric acid. When the first violent reaction is over, the flask is connected to a reflux condenser, and its contents are maintained in a state of gentle ebullition for four hours, after which they are allowed to cool and diluted with 50 c.c. of water. The next step is the reduction of the excess of chromic acid, of ferric salts, and of arsenic, and the distillation of the latter as trichloride. This may be accomplished by leading sulphur dioxide through the solution, adding 100 c.c. of fuming hydrochloric acid and 2 grms. of potassium bromide, saturating the mixture with hydrogen chloride, and distilling the arsenic trichloride in a current of hydrogen chloride and sulphur dioxide. The elimination of the sulphur dioxide from the distillate, however, by boiling under a reflux condenser and bubbling carbon dioxide through the liquid, is a tedious operation, and it is preferable to effect the reduction by means of cuprous chloride, of which at least enough is taken to reduce the excess of chromic acid, the whole of the ferric sulphate, and the arsenic. The cuprous chloride must not be added to the chromic acid mixture until the latter has been diluted, or chromyl chloride and free chlorine will result. When using cuprous chloride in place of sulphur dioxide to effect reduction, the estimation of arsenic in the distillate may be proceeded with at once. Arsenic is precipitated as sulphide in strongly acid solution, and the precipitate is filtered off, washed first with acid and then with water, dissolved in ammonia, the solution evaporated, and the residue oxidised by treatment with nitric acid, the excess of which is evaporated. The arsenic may then be estimated iodimetrically after reduction by means of sulphur dioxide and expulsion of the excess of the latter, but the authors prefer the following modification of the silver arsenate method: The arsenic acid remaining after evaporation of the nitric acid in the process last described is dissolved in water and transferred to a small beaker, the sulphuric acid also present is nearly neutralised with ammonia, 1 grm. each of ammonium nitrate and acetate are added, and then silver nitrate solution by small additions, until no further precipitation takes place. The precipitate is washed several times by decantation, and finally collected on a Gooch filter, washed with alcohol and ether, and dried at 120° C. Instead of being dried and weighed, the washed precipitate may be dissolved in dilute nitric acid and its silver content determined volumetrically by Volhard's method. The results thus obtained are from 1 to 4 per cent. lower than those yielded by the gravimetric method, but it is held that this is a quite satisfactory degree of accuracy.

G. C. J.

Action of Baryta Water on Certain Sorts of Glass. C. Bunge. (*Zeitsch. anal. Chem.*, 1913, **52**, 15-17.)—Baryta water (and also lime water) attack certain varieties of glass at the ordinary temperature. The crystalline scale which accumulated on the internal surface of a burette used for baryta was found to consist of barium sulphate, and it was proved that the sulphur was derived from the glass. The author suggests that manufacturers of laboratory glassware might give a guarantee that sodium sulphate was not a constituent of the batch. G. C. J.

Characteristic Reaction of Free Bromine. I. Guareschi. (*Atti R. Acad. Scienze*, Torino, 1912, 48; through *Chem. Zentralbl.*, 1983, I., 192-193.)—In previous communications (*Chem. Zentralbl.*, 1912, II., 635, 867), a solution of magenta or of Hofmann's Violet decolorised with sulphur dioxide was recommended as a sensitive reagent for free bromine (*cf.* Denigès, ANALYST, 1912, **37**, 581). Other decolorised dyestuffs may also be used for this purpose. Thus, Methyl Violet B Extra gives a less pronounced coloration than the normal dyestuff, whilst Methyl Violet (reddish) gives an intense violet coloration; and Crystal Violet gives a yellowish precipitate, which in the presence of excess of bromine changes first to green and then to blue. Indigo gives a blue coloration, which, however, is also produced by the action of atmospheric oxygen. By means of the magenta reagent it is possible to detect bromine in the ash of plants, beetroot, and in spinach. On acidifying a dilute solution of a hypobromite with hydrochloric or sulphuric acid, and adding the reagent, a blue coloration or reddish-violet precipitate (according to the concentration) is obtained.

In order to detect whether bromine is set free at high temperatures from organic compounds containing bromine, the vapours from the substance are conducted over filter-paper moistened with the reagent. A violet coloration indicates the liberation of bromine. C. A. M.

Estimation of Traces of Free Bromine. G. Denigès. (*Ann. Chim. anal.*, 1913, **18**, 8-11.)—The reagent previously described (ANALYST, 1912, **37**, 581) may be used for the estimation of traces of free bromine by taking advantage of the fact that an addition of hydrogen peroxide protects the bromine against the action of the sulphur dioxide. Two c.c. of the rosaniline and bisulphite solution (0·1 per cent. magenta solution, 1,000 c.c.; sodium bisulphite solution, sp. gr. 1·26 to 1·29, 10 c.c.; treated after five minutes with 20 c.c. of hydrochloric acid of sp. gr. 1·18 to 1·19) are shaken with 1 c.c. of chloroform and a small quantity of the liquid to be tested for bromine. If the chloroform layer becomes violet, colorimetric comparisons are then made with measured quantities of the solution and with standard solutions of free bromine, the reagent being previously diluted with an equal quantity of hydrogen peroxide solution. Test-papers prepared with this mixture are also more sensitive than those prepared with Schiff's reagent alone. C. A. M.

New Reagent for Bromine and Chlorine. G. Denigès and L. Chelle. (*Ann. Chim. anal.*, 1913, **18**, 11-15.)—The stock reagent is prepared by adding 10 c.c. of a 0·1 per cent. solution of magenta to 100 c.c. of 5 per cent. sulphurous acid, and allowing the mixture to stand until colourless. For the detection of free chlorine and bromine, 25 c.c. of the reagent are mixed with 25 c.c. of glacial acetic acid and 1 c.c. of sulphuric acid. Five c.c. of this mixture are shaken with 1 drop to several c.c. of the solution to be tested. In the presence of chlorine a yellow coloration is obtained, whilst bromine gives a reddish-violet colour, and the coloured compound in each case may be taken up with chloroform or carbon tetrachloride. The spectrum of the chloroform layer shows the two absorption bands (in the blue and the yellow) already described (ANALYST, 1912, **37**, 581), but the yellow chlorine derivative has a spectrum quite different from that given by Schiff's reagent with chlorine. In testing for the combined halogens, iodine is first eliminated by heating the sample with an iron persalt, and the bromine then liberated by means of sulphuric acid and potassium chromate. A glass rod with a pendent drop of sodium hydroxide solution is held in the vapours to absorb bromine, and then brought in contact with the mixed reagent and chloroform as described above. After elimination of iodine and bromine the substance is heated with potassium permanganate, and the liberated chlorine absorbed and detected in the same way as the bromine. In applying the test to the detection and estimation of bromine in sea-water, aqueous extracts of ignited organic matter, etc., 5 c.c. of the liquid are treated with 0·2 c.c. of hydrochloric acid of sp. gr. 1·18 to 1·19, 1 c.c. of concentrated sulphuric acid, 1 c.c. of the stock reagent, and 0·2 c.c. of a 10 per cent. solution of potassium chromate, and the mixture shaken after each addition, and, without cooling, shaken with 1 c.c. of chloroform. If the water contained as much as 0·001 grm. of bromine per litre, a violet to reddish-violet coloration is produced, and by comparing the colour with that given under the same conditions by standard solutions of bromides quantitative results are obtained. C. A. M.

Analysis of Cerium Compounds. E. Strohbach. (*Mitt. Techn. Versuchsamtes*, 1912, **1**, 45-56; through *J. Soc. Chem. Ind.*, 1913, **32**, 139.)—The method devised by the author is based on the fact that cerous salts are oxidised quantitatively to ceric salts by potassium chlorate in sulphuric acid solution, and that the results are not affected by the presence of hydrochloric or nitric acid or organic matter. If the prescribed conditions be adhered to, the method is quite safe, and it is stated that more than three hundred determinations have been made without the slightest accident. The oxidation is carried out in two stages in a modified Bangel distilling flask. This consists of a round-bottom flask of 7 cms. diameter, provided with a sealed-in air-inlet tube. The outlet tube leading from the neck of the flask first ascends and then descends, and is connected with a water-pump; it is provided with a safety-bulb. In carrying out the determination, 25 c.c. of the concentrated cerium chloride solution (about 200 grms. per litre) are treated with about 3 c.c. of dilute hydrochloric acid (1 : 10), to prevent precipitation of basic salts, and diluted to 500 c.c. Twenty-five c.c. of the diluted solution are placed in the distilling flask, together with 15 c.c. of potassium chlorate solution (a cold saturated solution), and a current of air, which has been led through cotton-wool and then through several towers fed with a concentrated solution of potassium permanganate, is forced through the mixture so as thoroughly to agitate it; 15 c.c. of concentrated sulphuric acid are now introduced, about 2 c.c. at a time, and the flask is connected with the pump. The open flask is warmed with a small flame, and when the main reaction is over a further quantity of 15 c.c. of sulphuric acid is introduced. The temperature should now be about 100° C., and the liquid should have become colourless. Ten c.c. of the chlorate solution are introduced in order to ascertain whether the cerium chloride is completely converted into sulphate; if this be not the case, the liquid becomes coloured yellowish-green by chlorine. The introduction of chlorate solution is repeated until the liquid remains colourless. The flask is now heated with a full flame until the thermometer registers 160° C., when the flame is removed, and the liquid allowed to cool to 100° C. Any deposit adhering to the stem of the thermometer or the neck of the flask is rinsed off with 1 to 2 c.c. of concentrated sulphuric acid. In this stage of the process most of the cerium chloride is converted into ceric sulphate. In the second stage the oxidation is completed, and the excess of chloric acid is decomposed. (Experiments have shown that chloric acid is decomposed completely at 150° C., whilst perchloric acid is not appreciably decomposed below 140° C.) A mixture of 10 c.c. of the chlorate solution and 15 c.c. of water is introduced into the flask, and the mixture is heated for two to three minutes at 133° to 135° C., then cooled to 90° C., the current of air being continued the whole of the time, diluted with cold water, cooled, rinsed into a beaker, made up to 400 c.c., and titrated with hydrogen peroxide solution (0·6075 grm. per litre, with the addition of 5 per cent. of sulphuric acid to increase the stability), a slight excess of the latter being added, and this titrated back with permanganate solution (1·1296 grms. per litre; 1 c.c. = 0·005 grm. of cerium). The reactions upon which the process is based are as follow:

$$4Ce_2(SO_4)_3 + 4H_2SO_4 + 3HClO_3 = 8Ce(SO_4)_2 + HClO_4 + Cl_2 + 5H_2O;$$
$$5HCl + HClO_3 = 3H_2O + 3Cl_2;$$
$$2Ce(SO_4)_2 + H_2O_2 = Ce_2(SO_4)_3 + H_2SO_4 + O_2.$$

Analysis of Mixtures of Hydrogen with Methane, Ethane, and Propane. P. Lebeau and A. Damiens. (*Comptes rend.*, 1913, **156**, 144-147.)—The method consists in subjecting the mixture to the temperature of liquid air and pumping off the hydrogen and methane, which are then separately estimated in the usual manner. The temperature of the vessel containing the liquefied ethane and propane is then allowed to rise, and these gases are transferred by means of a pump to another vessel in which they can be measured and separately estimated by simple calculation from the results of an explosion experiment. When methane is a minor constituent of the mixture, the exhaustion at the low temperature must be prolonged to something like two hours to obtain satisfactory results. The method greatly simplifies the analysis of mixtures of hydrogen with more than one hydrocarbon of the methane series, and is recommended in all cases where higher homologues of methane may possibly be present. G. C. J.

Separation of Iron and Manganese. F. H. Campbell. (*J. Soc. Chem. Ind.*, 1913, **32**, 3-4.)—The method is based upon reactions similar to those occurring in Stock's method for the precipitation of aluminium (*Comptes rend.*, 1900, **130**, 175). When a mixture of potassium iodide and iodate is added to a solution containing a ferric salt, the iron is completely precipitated, while manganous salts are unaffected in the cold. The cold solution of iron and manganese salts, preferably the sulphates, is nearly neutralised with ammonia, and a mixture of equal parts of a 25 per cent. potassium iodide solution and of a saturated solution of potassium iodate is then added. The precipitate is allowed to settle, the liberated iodine is removed by a slight excess of thiosulphate, the precipitate is then filtered cold and washed with water, the temperature of which is gradually increased to boiling. The filtrate is acidified with dilute hydrochloric acid, 5 c.c. of a saturated solution of ammonium phosphate added together with 20 grms. of solid ammonium chloride. Ammonia is added drop by drop until the solution is faintly alkaline; digestion at about 80° C. will then render crystalline the amorphous precipitate at first obtained. The precipitate is washed with very dilute ammonia, and ignited to manganese pyrophosphate. This method for dealing with the manganese was adopted because the presence of iodine renders inconvenient the ordinary methods depending on its oxidation to the manganic condition. When calcium or magnesium are present, the manganese should be separated as sulphide and then dissolved in acid, and precipitated as above. Nickel, cobalt, and zinc phosphates, being soluble in ammonia, do not interfere. It is important that the above directions are adhered to, or good results are not obtained. Tables are given showing the results of the method when applied to test solutions, the iron as a rule showing 0·1 mgrm., and the maganese 0·5 mgrm., high on the weights taken (0·02 to 0·2 grm.). H. F. E. H.

The Oxalate-Permanganate Method for Estimating Copper in Presence of Cadmium, Arsenic, Iron, or Lead. H. L. Ward. (*Zeitsch. anorg. Chem.*, 1912, **77**, 257-268.)—The best modification of this method, when copper has to be separated only from cadmium and quinquivalent arsenic, is to make the precipitation in a small bulk of 50 per cent. acetic acid. The solution should also contain from 5 to 10 per cent. of nitric acid, and precipitation is effected by adding to the boiling solution oxalic acid at the rate of 3·5 to 4 grms. for every 100 c.c. The precipitate is filtered off next day on asbestos, washed with no unnecessary excess of water, dissolved in 200 c.c. of hot 5 per cent. sulphuric acid, and titrated with permanganate. The results are always a little below the truth, but the maximum error is 1 mgrm. with 0·2 grm. copper present, and an equal or greater amount of cadmium or arsenic. Alkali salts in large amount do not interfere, but ammonium salts must be absent. Nearly as good results are obtainable without the use of acetic acid, but in that case it is necessary to evaporate the solution to dryness, and to follow closely the directions given for extracting the cadmium or arsenic from the residue. A modification of the evaporation method gives fair results in presence of not more than 0·1 grm. of iron, but the best method in presence of much iron is to make the solution just alkaline with caustic potash, and then to add twice its volume of acetic acid, heat to boiling, and add a large excess of oxalic acid (at least 4 grms. for every 100 c.c.). In this way 0·2 grm. of copper may be estimated with an error never exceeding 2 mgrms. in presence of 0·5 grm. of iron. Though lead oxalate alone is easily soluble in nitric acid, it is co-precipitated to some extent with copper oxalate. For the estimation of copper in presence of lead, therefore, the solution is mixed with an equal volume of acetic acid, the lead precipitated as sulphate by addition of 3 to 5 c.c. of sulphuric acid, and, with or without previous filtration of the lead sulphate, the copper is precipitated as oxalate and determined in the manner above described. The worst of thirteen test numbers given is 0·1527 copper found, with 0·1533 present.

G. C. J.

Estimation of Lead, Nickel, and Zinc, by Precipitation as Oxalate and Titration with Permanganate. H. L. Ward. (*Zeitsch. anorg. Chem.*, 1912, **77**, 269-274.)—Lead can be precipitated as oxalate from boiling acetic acid solution, and estimated by treating the washed precipitate with hot dilute sulphuric acid and titration with permanganate. Oxalic acid is a more effective precipitant than ammonium oxalate, and to obtain the most exact results the solution should contain 50 per cent. of acetic acid. Under these conditions 0·1 grm. of lead can be estimated with an error not exceeding 0·5 mgrm. Nickel and zinc can be estimated in a similar manner, but a slight modification is necessary to obtain a precipitate which can be filtered. The precipitation is made in boiling neutral aqueous solution, after which an equal volume of acetic acid is added and the mixture allowed to stand overnight before filtration. In the case of nickel, cobalt sulphate solution is added before the titration in such amount that the pink colour of the cobalt salt just predominates over the original green colour of the solution.

G. C. J.

Detection and Colorimetric Estimation of Lead, Copper, and Zinc, in Supply-Water. L. W. Winkler. (*Zeitsch. angew. Chem.*, 1913, **26**, 38-44.)—The sensitiveness of the hydrogen sulphide test for lead, whether in alkaline or acetic acid solution, is increased by the presence of ammonium chloride. The author tests two portions of the water as follows: To one portion (100 c.c.) are added 2 c.c. of 10 per cent. acetic acid, 2 grms. ammonium chloride, and 2 to 3 drops of 10 per cent. sodium sulphide solution. To another portion 2 to 3 drops of 10 per cent. potassium cyanide are added to convert ferrous iron to ferrocyanide and prevent the precipitation of copper as sulphide. Ammonium chloride (2 grms.) is added after two to three minutes, and followed by 5 c.c. of 10 per cent. ammonia and 2 to 3 drops of the sulphide reagent. In the absence of copper the two solutions are nearly equal in tint, but if both metals are present the acid solution is the darker, whilst if copper alone is present the alkaline solution is colourless. In quantitative comparisons, the trouble of preparing a number of standards may be avoided by having ammonium chloride present. In these circumstances exactly the same tint is obtained by running a standard lead solution from a burette into a sulphide solution as by adding the sulphide to an equal quantity of lead solution previously diluted.

In absence of lead, copper is best estimated colorimetrically as sulphide, again with the addition of ammonium chloride, which approximately doubles the intensity of the coloration. But in presence of lead it is estimated by the ferrocyanide method, after which the lead may be estimated as follows: A few drops of potassium cyanide solution (10 per cent.) are added to the water sample and to the copper standard, when the red colour gives place to a yellowish-green. This colour, which is equal in the two cylinders, does not interfere with the subsequent estimation of the lead as sulphide.

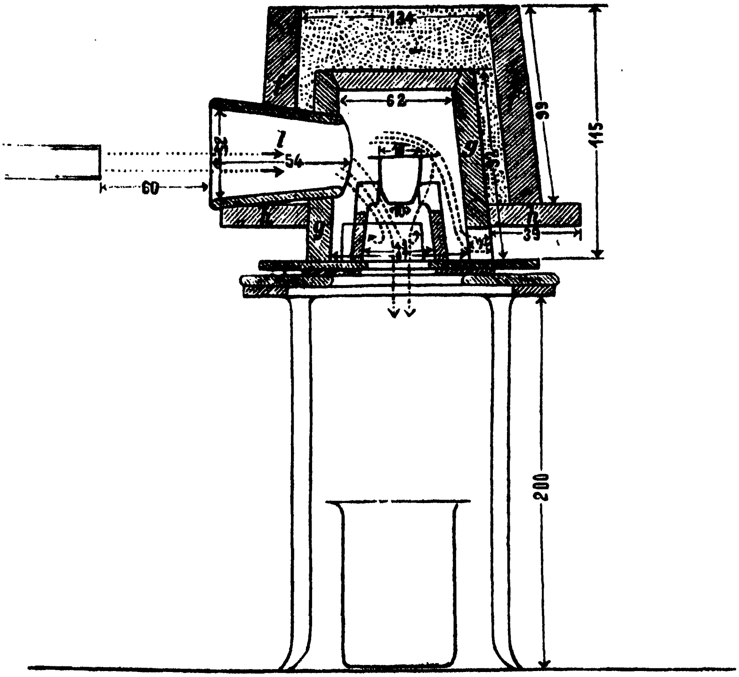
The paper also deals with the estimation of such minute amounts of lead, copper, and zinc, as can only be determined by concentrating to a small bulk the metal from large volumes of water. G. C. J.

Measurement of the Absorption of Oxygen by Sewage Effluents. H. J. S. Sand and S. R. Trotman. (*J. Soc. Chem. Ind.*, 1912, **31**, 1166-1167.)—The method of Letts and Adeney (ANALYST, 1909, **34**, 42) is based upon the rate at which oxygen from the air is absorbed by a sample of the effluent, special importance being attached to the rate. The authors find that this rate of absorption is incapable of accurate measurement in the manner described; what is measured being really the rate of agitation of the liquid, since the rate at which oxygen is absorbed depends, for a moderate degree of agitation, simply on the intensity of this agitation. The apparatus as supplied is unsuitable for putting in a shaking machine, and without such treatment no determination was ever found to reach completion, nor could concordant results be obtained. The "rate" of absorption measured was not found to bear any direct relation to the "chemical" absorbing power of the effluent, but was a measure of fortuitous convection currents in the liquid. It is recommended that the sample of effluent be agitated in a shaking machine for several hours in a bottle of about 300 c.c. capacity, which is closed with a rubber cork holding a tap. The amount of oxygen absorbed is then measured by connecting the bottle with a nitrometer of narrow bore, graduated in tenths of a c.c., which contains water. In this way, by neglecting the "rate" and measuring only the total amount of oxygen absorbed, reliable results are attainable. H. F. E. H.

Titration of Phosphoric and Boric Acids. W. Biltz and E. Marcus. (*Zeitsch. anorg. Chem.*, 1912, **77**, 131-136.)—In absence of calcium salts, phosphoric and boric acids may be estimated successively with fair exactness by making the solution just neutral to methyl orange, then titrating the phosphoric acid with caustic soda in presence of phenolphthaleïn ($NaOH = H_3PO_4$), and continuing the titration after addition of mannitol to determine the boric acid ($NaOH = H_3BO_3$). There is a tendency for the phosphoric acid to be over-estimated and for the boric acid to be underestimated. Contrary to statements current in textbooks (*e.g.*, Classen, "Massanalyse," 1912, 247), it is found that the presence of calcium and magnesium salts, even in large amount, is of no influence whatever on the estimation of boric acid by titration, provided phosphoric acid is not simultaneously present. The presence of magnesium salts has very little influence on the titration of phosphoric acid, and such effect as it has tends to counteract the normal error of a mixed phosphoric-boric acid titration. The influence of calcium salts on the titration of phosphoric acid is profound. The whole of the calcium is transformed into triphosphate (provided enough phosphoric acid is present) before the titration, with phenolphthaleïn as indicator, is complete. It follows that no titration of phosphoric acid in presence of calcium salts gives any measure of the phosphoric acid present, unless the amount of calcium present is accurately known. G. C. J.

Estimation of Potassium as Perchlorate in Potash Fertilisers. A. Strigel and J. Dodt. (*Landw. Vers. Stat.*, 1912, **78**, 179; through *Chem. Zentralbl.*, 1912, II., 2145.)—The chief source of error is the presence of hydrochloric acid, which converts perchlorates (*e.g.*, of barium or sodium) dissolved in alcohol into chlorides insoluble in alcohol. The evaporated mixture of perchlorates is hygroscopic, and the solubility of the potassium perchlorate is increased on exposure to air. The method proposed is as follows: Ten grms. of the salt are heated in a 0·5 litre measuring flask with about 300 c.c. of water and 2 c.c. of hydrochloric acid (sp. gr. 1·125), and boiled for five minutes; the sulphuric acid is precipitated with a small excess of barium chloride. When cool, the liquid is diluted to the mark and filtered; 25 c.c. of the filtrate are evaporated in a glass dish to a stiff syrup with 10 c.c. of 22 per cent. perchloric acid. The residue is treated in the usual manner, and the potassium perchlorate, collected on a Gooch crucible, is dried at 130° C.
O. E. M.

Opening up Silicates. W. Hempel. (*Zeitsch. anal. Chem.*, 1913, **52**, 86-90.)—If means exist for obtaining a temperature of 1,360° C., 1 grm. of felspar, topaz, or andalusite, can be decomposed by heating for ten minutes with 3 grms. of barium carbonate. One advantage of the method is that a crucible of 5 c.c. capacity and about 4 grms. in weight suffices for the purpose, an important consideration with platinum at its present price. Other advantages are the speed with which the decomposition is effected, and the ease with which the barium can be eliminated as sulphate. In deciding how much barium carbonate to take, regard must be had to Berzelius's observation that there is no risk of loss of alkalis, even at very high temperatures, provided the sum of the bases is less than that necessary to form orthosilicates. The necessary temperature is easily obtained by means of the electric furnaces now procurable, but can also be obtained by means of a blowpipe and the furnace illustrated. The cross-hatched portions represent plumbago parts, the dotted field a packing of kieselguhr, and the dimensions are given in millimetres.



G. C. J.

Mineralogical Analysis of Soils. W. H. Fry. (*J. Ind. and Eng. Chem.*, 1913, 5, 30-32.)—In this paper the mineralogical analysis of soils is advocated as leading to useful results and enabling a judgment to be passed on their probable origin and fertility. It is possible to rapidly and accurately ascertain the presence of a large number of rock-forming minerals chiefly by microscopical petrographical methods and by the use of dense solutions such as methylene iodide, while by employing a series of liquids of known refractive index this constant can also be determined for the soil particles under examination. The limitations of the microscope have so far prevented the mechanical examinations of the soil fractions other than the sands and the silts; the clays, on account of the extreme minuteness of their grains, do not permit of microscopic identification. A series of soil analyses is given; thus, in a sandy loam from Orangeburg there were found magnetite, quartz, oligoclase, microline, orthoclase, oligoclase-albite, labradorite, biotite, garnet, zircon, epidote, apatite enclosed in quartz, and muscovite. The proportion of ferric minerals was very low. The method shows definitely in what form the elements are combined, a result incapable of being obtained by chemical methods, and it is possible that it may be the means of constructing a mineralogical classification of soils. H. F. E. H.

Significance of the Lime-Magnesia Ratio in Soil Analyses. P. L. Gile and C. N. Ageton. (*J. Ind. and Eng. Chem.*, 1913, **5**, 33-35.)—Loew's hypothesis that plants only make their maximum growth when the lime-magnesia ratio is at an optimum (from 1 : 1 to 4 : 1), though widely tested, has not always been confirmed. Analyses are quoted showing that the good pineapple soils of Porto Rico have unusually wide lime-magnesia ratios, that an exceptionally fertile soil has a ratio of 25 to 1, and that some soils ranging up to 500 to 1 are at least ordinarily productive. With the sugar-cane soils of the same country no correlation between the fertility and the lime-magnesia ratio could be established, exceptionally productive soils showing variations of from 22 to 1 up to 1,460 to 1. Single salt solutions are well known to exert a toxic influence on plant growth, which is negatived when they are present together, lime salts in this relation having a powerful antagonistic action on the toxicity of magnesium salts, but it does not follow that such an antagonism is exerted in the ordinary soil complex. The authors conclude that in analyses of ordinary soils, the lime-magnesia ratio is of no significance, but in analyses of the soluble salts of alkali soils the ratio may be important, since in such soils there are relatively concentrated solutions of soluble salts. Direct experiments on growing plants gave conflicting results, and it is probable that the apparently confirmatory results arrived at by others are to be explained by alterations in the sensitive factor of soil reaction, and not to the lime and magnesia *per se*. H. F. E. H.

Determination of Stannous Chloride and of Chlorates by Means of Methylene Blue. F. W. Atack. (*J. Soc. Dyers and Col.*, 1913, **29**, 9-10; through *J. Soc. Chem. Ind.*, 1913, **32**, 163.)—Solutions of stannous chloride, made strongly acid with chlorine-free hydrochloric acid, are titrated in an atmosphere of carbon dioxide with standardised $\frac{N}{50}$ methylene blue solution (*i.e.*, 4 grms. per litre). Below a certain concentration of acid no reduction of the methylene blue occurs, and a suitable concentration is obtained by adding an equal volume of concentrated hydrochloric acid to the portion of stannous chloride solution to be titrated. When checked with a sample of pure tin, results obtained were identical with those for the gravimetric estimation. Antimony, iron, zinc, manganese, lead, bismuth, and aluminium, do not affect the process; but the method cannot be used in the presence of copper, titanium, tungsten, molybdenum, or vanadium. The methylene blue solution may be standardised in the usual way with titanous chloride (Knecht, *J. Soc. Chem. Ind.*, 1905, **24**, 154) or by reducing with titanous chloride solution, adding a portion of a solution of known strength of pure potassium chlorate (about 0·7 grm. per litre), then reducing the methylene blue formed with titanous chloride, and so finding the value of the methylene blue solution in terms of oxygen of the pure potassium chlorate solution. Where titanous chloride is not available, stannous chloride can be used, and these two processes constitute rapid methods for the estimation of chlorates. When methylene blue solution is slowly added to potassium perchlorate solution, a quantitative precipitation of the perchlorate takes place, and this is filtered off, washed, dried, and weighed. These four methods for the standardisation of methylene blue solution give results which agree very well.

Estimation of Sulphurous Acid in Air. C. Kullgren. (*Arkiv för Kemi, Min. och Geol.*, 1912, **4**, No. 31; through *Chem. Zentralbl.*, 1912, II., 2145.)—In the course of conductivity determinations on dilute solutions with a Kohlrausch conductivity cell, it was found that results were sometimes affected by sulphur dioxide present in the air used to dry the cell, and absorbed by the platinised electrodes. This was made the basis of a method for the estimation of small quantities of sulphur dioxide. The sample of air under examination is repeatedly passed backwards and forwards through spongy platinum, and the sulphuric acid formed dissolved out and determined by methods described at length in the original. The result is not affected by the presence of ammonia. A pipette for measuring and mixing small quantities of gas, and a portable gas sampler, are described.

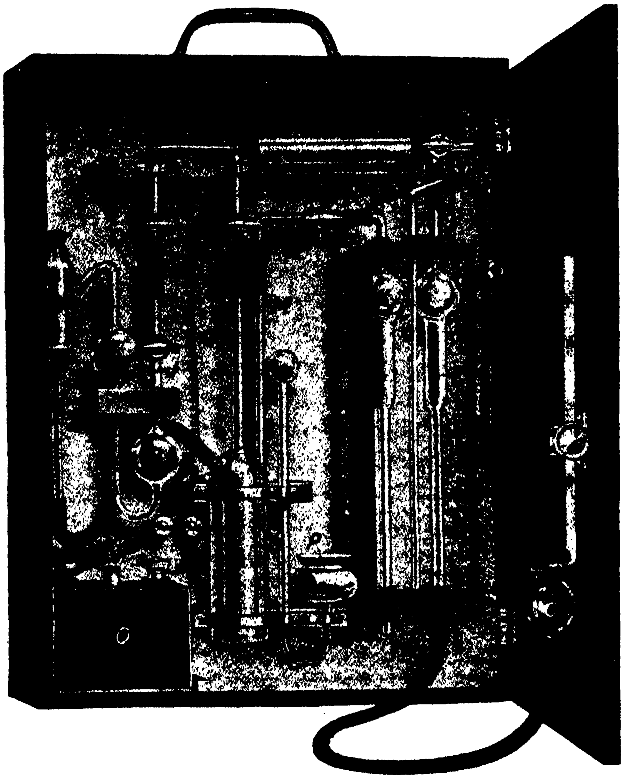
O. E. M.

Use of Potassium Palmitate in Water Analysis. C. Blacher, P. Grünberg, and M. Kissa. (*Chem. Zeit.*, 1913, **37**, 56-58.) — Potassium palmitate solution is recommended for use in the estimation of hardness in water, of sulphates in the same, etc. One hundred c.c. of the water are first titrated with $\frac{N}{10}$ hydrochloric acid, a drop of a saturated (1 per cent.) alcoholic dimethylaminobenzene solution being added as indicator, the colour change of the latter being from yellow in alkaline solution to red in acid solution; during the titration the liberated carbon dioxide must be removed by blowing a current of air through the water. Standardised potassium palmitate solution is then added until the mixture becomes faintly alkaline in reaction towards phenolphthaleïn. The quantity of acid required corresponds with the carbonate hardness of the water, and the amount of palmitate solution with the total hardness. For the estimation of sulphates the water is neutralised, treated with a definite volume of $\frac{N}{10}$ barium chloride solution, filtered, and the filtrate titrated with potassium palmitate solution; the quantity of the latter used is equivalent to the total hardness plus the excess of barium chloride, and the amount of sulphate present may then be calculated. W. P. S.

Rapid Method of Estimating Zinc. K. Voigt. (*Zeitsch. angew. Chem.*, 1913, **26**, 47-48.) — The method described by the author (ANALYST, 1912, **37**, 35) has been criticised by Hassreidter, who, however, did not adhere to the author's directions (*ibid.*, 1912, **37**, 153). Those directions were arrived at empirically, but it is now shown that the excellent results yielded by the method are dependent on the presence of large quantities of ammonia and ammonium chloride. Hassreidter's low results were due to his employing very much less ammonia than the author recommended. Equally unsatisfactory results attend the use of minimum quantities of acid for solution of the ore or the evaporation of much of the excess of acid. In such a case there will be insufficient ammonium chloride present unless 30 c.c. of a saturated solution of the salt be added. If the quantities named in the original paper be employed, excellent results are obtained. The function of the ammonia and ammonium chloride is to inhibit re-formation of zinc silicate. G. C. J.

APPARATUS, ETC.

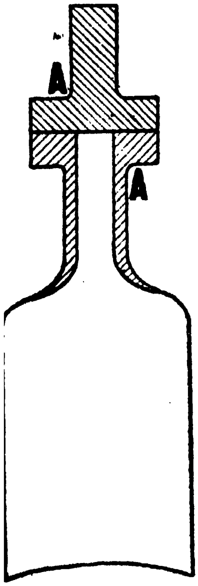
Apparatus for the Examination of Mine Air. L. A. Levy. (*J. Soc. Chem. Ind.*, 1913, **31**, 1153-1155.)—A special form of silica-platinum oxidation capillary-tube is described suitable for the estimation of small quantities of methane, carbon monoxide, and hydrogen, in air and in other gases. The tube can be employed in ordinary gas analysis apparatus as well as in the two special instruments for mine air examination described in the present paper. Sections of the Coal Mines Act (1911) calling for the routine examination of the air of mines are quoted, which show that the provisions of the Act call for two separate instruments—one for the rapid and accurate estimation of fire-damp, and the second for the estimation of carbon dioxide, oxygen, and preferably also of fire-damp. A special form of apparatus (illustrated) for each of these two purposes is described in detail, embodying, among other improvements, the silica-platinum capillary just described. Both instruments are small,



compact, and readily portable. The salient points of the second and larger apparatus, which weighs 8 pounds complete with mercury, battery, etc., are—(1) The oxidation of the inflammable gases in the special silica-platinum tube (*F*), which is 0·5 mm. bore. The ignition is effected by a platinum wire fused into the walls, a current of 2·5 ampères from a 2-volt cell being employed. The fine bore of the leading-in tubes acts as a valve, and prevents the passage of the gas over the heated wire at too great a speed. It appears probable that improved working will be effected by coating the interior walls of the silica tube with finely divided platinum or palladium. (2) The measurement of the gas in a special burette, giving reading of adequate accuracy without undue length of stem. (3) Safety devices rendering the instrument safe in use. (4) A correction tube compensating for slight changes in temperature and pressure during the analysis. (5) Means for the rapid and complete absorption of oxygen.

The gas to be analysed is drawn into the burette *E*, through the three-way cock *D*, by means of the mercury reservoir *C*. The capacity of the burette is 25 c.c. It consists of—(*a*) A narrow graduated stem reading up to 4 per cent., and divided to 0·02 per cent. The percentages of carbon dioxide and methane are observed on this stem. (*b*) A broader graduated portion reading up to 22 per cent., and divided to 0·2 per cent. The oxygen is read in this portion. (*c*) An ungraduated bulb.

The connection-tube *K* is of the same shape and capacity as the burette, and communicates with the vertical capillary-tube through the ventcock *L*. The burette *E* communicates through the water-jacketed oxidation capillary *F* to the potash pipette *A*. The burette can also be joined up with the absorption pipette *B* through the cocks *O* and *J*. Before commencing an analysis, the potash in the vessel *A* is filled up to the mark on the capillary neck at *G*, and the alkaline pyrogallate is filled up to the mark at *J*. The sample of gas is drawn into the burette, the mercury is adjusted to the zero by the rack and pinion, and the cock at *H* is opened. The potash levels are adjusted in the tubes *G* and *M* by raising or lowering the potash reservoir *N* attached to the vessel *A* by the rubber tube. The cock *L* is closed. The gas is passed into the potash pipette, and back again into the burette, adjusting the potash levels as described. The reading of the mercury level in the burette gives the percentage of carbon dioxide. Current is passed through *F* by depressing the leather top *P* of the mercury switch, the mercury thus rising and completing the circuit. The gas is passed over the heated wire into the potash and back again two or three times. The percentage of methane is equal to one-third of the contraction now observed. The cock *H* is closed, and the gas is passed into the pyrogallate pipette *B*. The cocks *O* and *J* are closed, the pipette *B* is detached and shaken, and the oxygen thus rapidly absorbed. The pipette is reattached, and the gas drawn back into the burette. The mercury reservoir is adjusted until the pyrogallate reaches the mark on the capillary neck at *J*. Twice the percentage of methane when added to the contraction now observed gives the amount of oxygen. This observation can be made with an accuracy of 0·1 per cent. Before each observation the water in the jacket is mixed by blowing through the tube *R*. With the first apparatus, which is designed for the estimation of methane only, results are recorded dealing with analyses of artificial mixtures of air and methane, this latter gas being capable of estimation with an accuracy of 0·01 per cent. H. F. E. H.



Improved Pyknometer for Density of Solids. J. Johnston and L. H. Adams. (*Chem. News*, 1913, **107**, 56.)—The essential feature of the pyknometer illustrated is the plane ground joint between stopper and bottle. The neck is made thick, not only to give strength, but to minimise heat transfer when the bottle is held by the neck between the fingers. The surfaces making up the joint should be *optically flat*, but not necessarily highly polished. As a criterion of the quality of the joint, the stopper should be pressed forcibly on to the bottle with a slight rotatory motion, after which it should be possible to lift the bottle by the stopper. In making this test, the stopper should be dry, without grease or other lubricant. Another requisite of success is that the pyknometer should be made in such a manner that no deep groove exists at *A*, or, indeed, that there be no recess from which excess of water cannot readily be wiped away. The advantages of the instrument are that the loss in weight by evaporation is negligible (1 mgrm. water in twenty-four hours), no grease or other lubricant is required, and any small particles of the solid under examination which may accidentally lodge on the ground surface can be easily wiped off. By the use of a thermostat controllable within 0·01° C., duplicate weighings did not differ by as much as 0·2 mgrm., but the capacity of the bottle is not stated. Filled with water, it weighed 30 grms., and, if the illustration is full size, its capacity must have been 20 c.c. or a little less. G. C. J.

Micropyrometer. G. K. Burgess. (*J. Wash. Acad. Sci.*, 1913, **3**, 7-10; through *J. Soc. Chem. Ind.*, 1913, **32**, 162.)—An apparatus is described for determining through a microscope the melting-points of minute specimens (0·001 mgrm. or less). This is constructed by mounting in the Huyghens eyepiece of a microscope a small incandescent lamp which is in series with a rheostat and ammeter. The substance is placed upon a strip of platinum foil, electrically heated, and the tip of the filament of the lamp is set to the same brightness as the platinum strip, viewed from above at the instant of melting of the specimen on which the microscope is focussed. Thus the specimen, the platinum strip, and the lamp filament are seen all in focus at once, and the current passing through the filament is taken as a measure of the temperature of the strip. In practice, the observer with one hand raises the temperature of the strip by increasing the current through it by means of a fine-step rheostat, and with the other hand adjusts the current through the lamp by means of the other rheostat. The eyepiece is covered with a slip of monochromatic glass. For temperatures above which the lamp should not be heated (about 1400° C.), an absorption glass is placed between the microscope objective and the window of the furnace. In the instrument constructed at the United States Bureau of Standards, the microscope has a 48 mm. single achromatic objective and a 6·4 × eyepiece. The tubular furnace in which the platinum strip is situated has a circular window immediately over the strip; the interior is blackened, to prevent reflection, and the exterior is cooled by a blast of air. Of the methods for calibrating the pyrometer, the more convenient and accurate consists in observing the melting-points of two or more known pure substances. Certain errors are thus eliminated, although it is not permissible to deduce results for unknown substances of one kind (metals or salts) from calibrations made with substances of a different kind. The combination of microscope and pyrometer enables changes occurring at the surface of refractory substances to be observed at various temperatures.

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REPORTS.

Report of the Acting Chemist for 1912, U.S. Department of Agriculture. R. E. Doolittle.—The Report consists of a brief outline of the work done in a large number of special departments, and covers a very wide field. Practically every subject upon which the chemist can help the agriculturist has received attention, and in addition a large number of special investigations too numerous to record in detail are summarised.

An outline is appended of the special work planned for the fiscal year 1912-13, food preservation in its many branches taking an important position. It is interesting to note that considerable difficulty has been experienced by the Bureau of Chemistry and branch laboratories in obtaining certain special reagents of the required degree of purity; among these may be mentioned "special" acetic acid 99·9 per cent. purity, and hydrochloric acid and zinc free from arsenic. The whole Report bears witness to the elaborate organisation and sound work devoted to the inspection and chemical analysis of food products in the United States.

H. F. E. H.

Report on the Work of Inspectors of Foods for the Year 1911-12. A. W. J. Macfadden. (Extracted from the Annual Report of the Medical Officer of the Local Government Board.)—The Report is arranged under the following headings: (1) Work in Special Relation to Public Health (Regulations as to Food) Act, 1907; (2) Local Arrangements for Food Inspection; (3) Bacterial Food-Poisoning; (4) Work in Relation to the Sale of Food and Drugs Acts.

I. The work under the regulations has proceeded satisfactorily, and at certain ports additional assistant officers have been appointed. Improvement continues to be maintained as regards the soundness and freedom from disease of meat imported from abroad, the difficulties as regards inspection on importation of Australian beef affected with *onchocerciasis*, referred to in the Annual Report for last year, having been lessened considerably in consequence of the greater attention given to examination of the meat in Australia. Improvement in the condition as regards disease of offal (livers, kidneys, lungs, etc.) has not been so satisfactory.

The authorities continue to pay special attention to Continental meat coming from German ports. The formaldehyde process of treating refrigerated meat has been discontinued in the case of meat from Australia, and at present appears to be confined to meat arriving from Argentina. During the year samples were taken on five occasions, and in all but one of them analyses showed that the importers had maintained the improvement previously noted in regard to the amount of formaldehyde taken up by the meat.

Unsound Food Regulations.—A consignment of fruit pulp was found to contain a rather large amount of tin and over 30 grains of salicylic acid per pound.

The trade of imported milk from the Continent has been kept under observation, samples of this material, as well as butter and cream, having been examined in the Board's pathological laboratory for tubercle bacilli. All the milk samples had been pasteurised and reached a good standard of cleanliness, and in no case was *B. tuberculosis* discovered. Of the thirty-two samples of cream taken between July and December, 1911, only one, imported from Harlingen, and labelled "Preserved," was found to contain *B. tuberculosis*, and in no case was its presence detected in the forty-eight samples of imported butter. The packets of this material, when coming from the Commonwealth, New Zealand, Holland, and Denmark, bore labels indicating official inspection in the country of origin.

II. *Local Arrangements for Food Inspection.*—The arrangements provided locally for the inspection of meat and other food materials continue to be good in the larger cities and towns, but in many rural districts cannot be considered satisfactory. A more thorough system of inspection and control is urgently needed in many of the rural districts—for instance, Somerset, where a considerable traffic in diseased meat is known to have been carried on for some years. The matter of metallic contamination of oysters is receiving the Board's attention, results of recent examinations made at Liverpool and at the Government Laboratory having shown that in some cases zinc was present on an average in an amount more than double that of the copper found.

III. *Bacterial Food-Poisoning.*—In view of the importance of this subject, the Board issued a memorandum (given in full as Addendum B in this Report) in September, 1911, in which are indicated the steps to be taken in dealing with the investigation and prevention of outbreaks of this nature. The two most serious outbreaks during the year occurred at Chesterfield and Bacup. In the former case 161 persons were affected, and one death occurred, due to the consumption of pork pies infected with *B. suipestifer*. Owing to the fact that the local officers at Bacup were not successful in obtaining any of the original material which was suspected of having caused illness, the results of the investigation were inconclusive. Two hundred and thirteen cases and five deaths occurred, and the case illustrates the importance of at once obtaining samples of what remains over of the food actually eaten.

IV. *Work in Relation to the Food and Drugs Acts.*—It is recommended that local authorities should send to the Board copies of all special reports made by the public analyst, as in some cases, notably one in the county of Cheshire, valuable reports on some special investigation have escaped the knowledge of the Board.

During the year inquiries were completed and reports were issued on several important questions affecting administration under the Sale of Food and Drugs Act: (1) Bleaching and Other Chemical Treatment of Flour. Food Report No. 12 (ANALYST, 1911, **36**, 254); Food Report No. 19 (*ibid.*, 1912, **37**, 596). (2) Baking-Powder and Self-Raising Flour. Food Report No. 13 (*ibid.*, 1911, **36**, 255). (3) Bread. Food Report No. 14 (*ibid.*, 1911, **36**, 479-483). (4) Infants' Foods. Food Report No. 15 (*ibid.*, 1911, **36**, 572-573). (5) Preservatives in Milk and Cream. Food Report No. 17 (*ibid.*, 1912, **37**, 155).

Since the publication of this Report it has come to the notice of the Board that a further circular letter has been addressed to milk-vendors by the proprietors of "Mystin" (ANALYST, 1912, **37**, 155 and 178), stating that they have perfected a new preservative which will be invoiced simply as "No. 2," and which will only be supplied "to those of our old customers upon whose discretion we can rely," at the old price of 10s. 6d. per gallon. A sample has been examined by Dr. Monier-Williams, and was found to consist of a solution of potassium chlorate (5·93 per cent.) and formaldehyde (0·38 per cent.). The former chemical is apparently added under the impression that its presence will interfere with the tests for formaldehyde in the milk. This is not the case. The activity being shown in the sale of such preparations should emphasise the importance of vigilance on the part of local authorities in these matters.

Following upon the issue of Dr. Hamill's report on vinegar in 1908 (Food Report No. 5) representations from local authorities and traders were received by the Board, urging the desirability of statutory definition in regard to vinegar. While not at present possessing powers enabling them to make such definitions, the following definitions were suggested for the guidance of those concerned:

Vinegar is defined as a liquid derived wholly from alcoholic and acetous fermentations. It shall contain not less than 4 grms. of acetic acid (CH_3COOH) in 100 c.c. of vinegar. It shall not contain arsenic in amounts exceeding 0·0143 mgrm. per 100 c.c. of vinegar, nor any sulphuric or other mineral acid, lead, or copper, nor shall it contain any foreign substance or colouring matter except caramel.

Malt vinegar is to be derived wholly from malted barley or wholly from cereals the starch of which has been saccharified by the diastase of malt.

Artificial vinegar is any vinegar or substitute for vinegar containing, or derived from any preparation containing, any added acetic acid which is not wholly the product of alcoholic and subsequent acetous fermentation. It is to contain not less than 4 grms. of acetic acid (CH_3COOH) in 100 c.c. of the artificial vinegar. It is not to contain arsenic in amounts exceeding 0·0143 mgrm. per 100 c.c. of artificial vinegar, nor any sulphuric or other mineral acid, lead, or copper, nor is it to contain any foreign substance or colouring matter except caramel. H. F. E. H.

REVIEW.

ANALYSIS OF METALLURGICAL AND ENGINEERING MATERIALS: A SYSTEMATIC ARRANGEMENT OF LABORATORY METHODS. By HENRY WYSOR, B.S. Easton, Pa.: The Chemical Publishing Company. 1912. London: Williams and Norgate. 82 pp., with figures. Price $2·00.

This excellent book, and especially the introduction and the general notes on the care of apparatus, sampling, etc., will be found particularly useful to the student, for whom it is primarily written. The methods of analysis given are mainly selected for students, but, on account of the large field covered in order to meet the requirements of a general course for them, many methods now largely used by the technical man have been omitted—for instance, the bismuthate and persulphate methods for manganese.

As the author asks for criticisms and suggestions on this first edition, the reviewer would suggest the addition of an index and the dating of the table of atomic weights, and that the author might point out that, although the gravimetric factors for calculations, given on p. 80, are near enough for all practical purposes, they have not been calculated from the atomic weights as given. The atomic weight for iron, for instance, is given in the table as 55·84, but for calculating out the factors and for the example given on p. 10, the figure 56 has been taken—*i.e.*, the factor of 0·7000 for Fe to Fe_2O_3, instead of 0·6994, which would be the literally correct one calculated from the "latest" atomic weight. Such matters are, of course, of no practical importance, but a student who followed the advice given on p. 10, that he should calculate out his own factors, might be surprised to find that his results frequently differed from those actually given in the book.

As a handbook for the common-sense training of students who intend to go in for technical, commercial, or metallurgical work, it can be highly commended, and for those actually engaged in technical work, there are many useful hints which deserve attention.

The interleaving of the book with blank pages for notes is a useful feature, and the illustrations are particularly clear.

G. T. HOLLOWAY.

APRIL, 1913. Vol. XXXVIII., No. 445

THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

THE monthly meeting of the Society was held on Wednesday evening, March 5, in the Chemical Society's Rooms, Burlington House. The President, Mr. L. Archbutt, F.I.C., occupied the chair.

The minutes of the previous ordinary meeting were read and confirmed.

Certificates of proposal for election to membership in favour of Messrs. A. C. Bescoby, M.A., T. W. F. Clark, F. E. Day, B.Sc., A.I.C., and J. F. Tocher, B.Sc., F.I.C., were read for the second time; and certificates in favour of Messrs. Henry Francis Everard Hulton, A.I.C., 15, Oakhill Court, Putney, S.W., assistant chemist to Messrs. Watney, Combe, Reid and Co., Ltd.; Charles Thomas Kingzett, F.I.C., Newlands, Weybridge; and Alfred J. Parker, 1, Bulwer Street, Herne Bay, Auckland, New Zealand, public analyst for the City of Auckland, N.Z., were read for the first time.

Messrs. J. A. Goodson, F.I.C., F. W. Skevington, and J. C. White, A.I.C., were elected members of the Society.

The following papers were read: "The Bacterial Testing of Disinfectants: a Practical Criticism," by C. T. Kingzett, F.I.C., and R. C. Woodcock, F.I.C.; "A Quick and Improved Method for the Estimation of Boric Acid in Milk and Cream," by Frederic W. Richardson, F.I.C., and William Keighley Walton; "The Combined Estimation of Boric Acid and Fat in Cream," by Frederic W. Richardson, F.I.C.; "A Simple All-Glass Extraction Apparatus," by Clayton Beadle and Henry P. Stevens, M.A., Ph.D., F.I.C.; and "The Accurate Determination of Carbonic Acid in Carbonates," by Frank Sturdy Sinnatt.

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THE DETERMINATION OF CARBON DIOXIDE IN CARBONATES.

By FRANK STURDY SINNATT.

(*Read at the Meeting, March* 5, 1913.)

The difficulties encountered in the accurate determination of carbon dioxide evolved in the course of chemical reactions, particularly when the evolution of the gas takes place over an extended period, or when the amount of carbon dioxide produced is very small, will, it is hoped, justify the publication of the process described in this paper. The method appears to be applicable to many of the reactions which lead to the evolution of carbon dioxide; but for the purposes of comparison and as a guide to the quantities of reagents necessary, the estimations given in this paper have been carried out upon pure carbonates.

Of the many processes for the determination of carbon dioxide in carbonates, the following are in general use:

Schrödters' apparatus, or some modification, may be expected to yield results within 0·3 to 1 per cent. when the weight of substance available is about 1 grm. and the substance is rich in carbon dioxide.

An improved form of Scheibler's apparatus is described by S. H. Collins (*J. Soc. Chem. Ind.*, 1906, **25**, 518). This process depends upon the measurement of the volume of carbon dioxide evolved from carbonates by the action of acids. The method suffers from inaccuracies inherent in any process in which it is necessary to measure small volumes of carbon dioxide—viz., the effect of temperature and the impossibility of standardising the volume of gas remaining dissolved in the reaction liquid. The equivalence of six sources of error is given as 1 per cent. This method has a great advantage in that it is very rapid.

Hall and Russell (*J. Chem. Soc.*, 1902, **81**, 83) observe that the determination of small quantities of carbonates in such a material as soil is attended with many difficulties, and they state that when the soil contains only 0·5 per cent. of calcium carbonate the whole of the carbon dioxide produced, when acid is added, remains dissolved in the acid used for the decomposition of the carbonate. These authors describe a process whereby the volume of gas evolved by the action of sulphuric acid on a carbonate may be measured with accuracy.

The method in general use for the accurate determination of carbonates consists in decomposing the carbonate with acid, and, after purifying the gas evolved, to aspirate it through weighed potash bulbs. This process is inaccurate when the weight of carbon dioxide obtained is very small, unless the precaution of counterpoising the potash bulbs with an empty bulb be adopted. Even with this precaution variations in weight of the potash bulbs are possible from external causes.

Methods in which standard barium hydrate solution is placed in wash-bottles re accurate under certain conditions, but the following reasons have prevented them ing more generally adopted: The comparatively slight solubility of barium hydroxide vater, and the necessity of using a large volume of solution, and consequently a cumbersome wash-bottle; the fact that carbon dioxide is not absorbed by barium hydroxide solution very rapidly, and hence a slow stream of gas is essential; the prevention of contact with air and the breath of the experimenter during the work; the difficulty of titrating the barium hydroxide solution accurately when in the wash-bottle at the completion of the absorption.

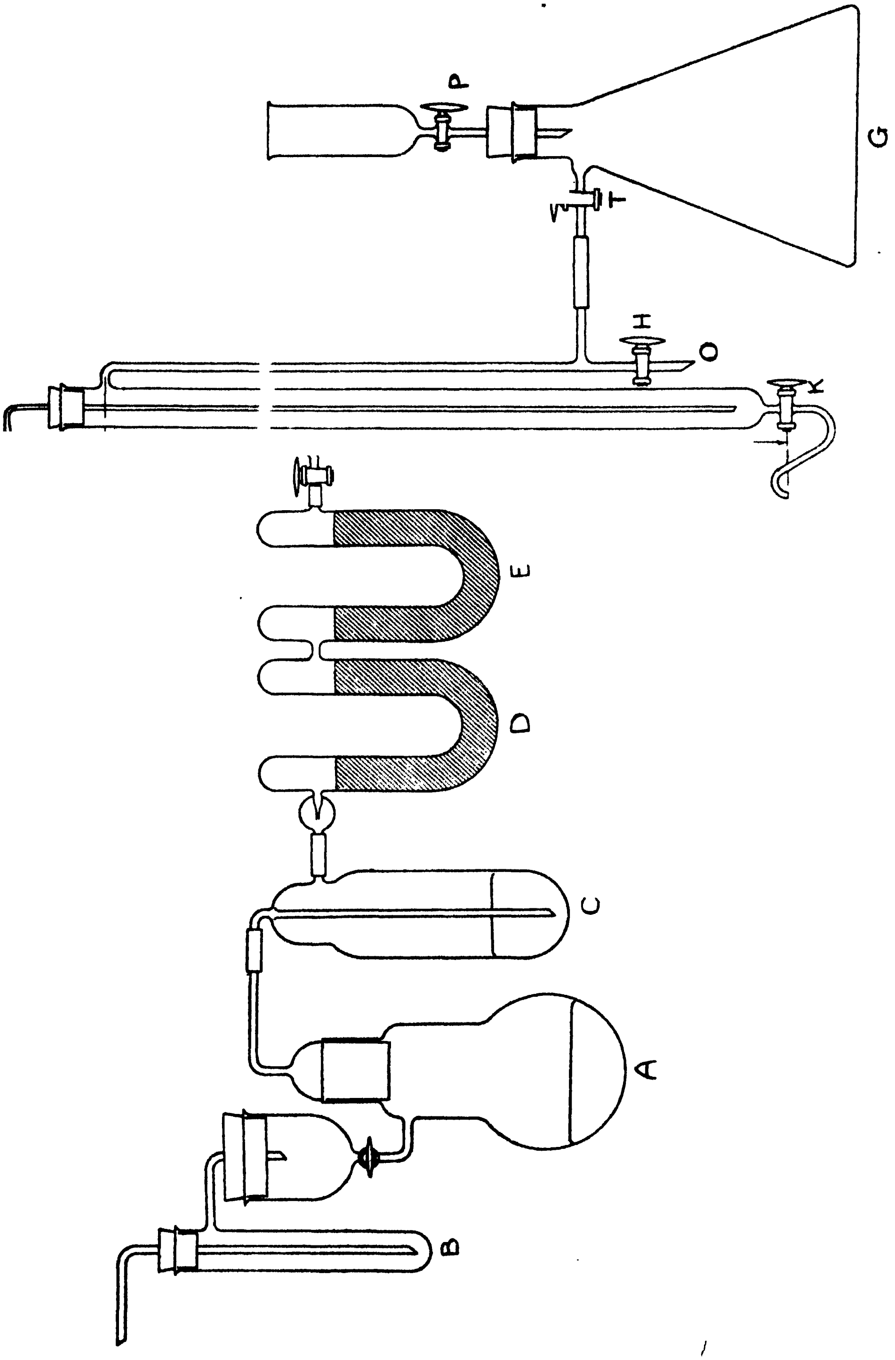
In a paper (ANALYST, 1912, **37**, 12) the author described an apparatus by means of which the rate at which a gas flowed into an evacuated vessel could be accurately and easily controlled. It is found that the carbon dioxide evolved from carbonates may be conveniently estimated by means of this apparatus.

The figure shows the complete train of apparatus employed for the determination of carbon dioxide in carbonates. *A* is the decomposition flask described by Lunge and Marchlewski. *B* is a wash-bottle containing caustic potash solution. *C* is a wash-bottle containing concentrated sulphuric acid. *D* a calcium chloride tube. *E* is packed with alternate layers of glass-wool and pure silver sulphate (to absorb hydrochloric acid gas). *F* is the special apparatus (*loc. cit.*). The flask *G* is of the usual type used for filtering. The capacity of the flask was 2·5 litres, but in some of the analyses one of a litre capacity was used. It is evident that the capacity of the flask limits the volume of air available for aspirating through the apparatus. The flask and the special apparatus should be freed from moisture previous to commencing an estimation.

The determinations were carried out in the following manner: The flask and the apparatus are filled with air freed from carbon dioxide. (If the percentage of carbon dioxide in the air be known accurately, a correction may be made for the carbon dioxide remaining in the apparatus and flask after the evacuation.) Taps *K*, *M*, and *P* are closed, *H* and *T* are opened, and *O* connected to a water-pump, and the whole evacuated. When the apparatus is exhausted, *H* is closed. A reservoir containing mercury is brought under the end of *F*, and mercury allowed to flow into the tube until no further quantity will enter. The tap *K* is then closed. The mercury column in the tube serves as a manometer, and shows the degree of exhaustion in the flask. A weighed quantity of the carbonate is shaken into the flask *A*, and an excess of hydrochloric acid is brought into the cup. The tube to the wash-bottle *B* and the ground stopper of *A* are then replaced. The tap *M* is now opened, and mercury is allowed to flow slowly from the tube into the reservoir by opening *K*; at the same time the hydrochloric acid is run cautiously into the carbonate. When the whole of the acid has been added, the communication between the cup and the bulb *A* should remain open.

The carbon dioxide evolved is aspirated into the flask; air is also drawn in, passing through the wash-bottle *B*, and carrying the carbon dioxide into the flask. The current of air need not be interfered with from this period until the determination is finished. The carbon dioxide remaining in solution in the liquid in the vessel *A* is expelled by slowly heating the contents, and then boiling gently for some time.

Air is permitted to flow through the apparatus until the pressure has fallen to about 700 mm., when the tap *T* into the flask is closed and the latter disconnecte from *F*. A measured volume of standard barium hydroxide solution is placed in t tap funnel, and allowed to enter the flask as quickly as possible; owing to



ntents of the flask being under diminished pressure, no gas is displaced by the barium droxide solution. The last traces of barium hydrate solution are washed from the

tap funnel with water. Air free from carbon dioxide is now passed into the flask until the contents are at atmospheric pressure. The estimation is then completed by the usual procedure adopted for Pettenkofer's process. The time required for a determination was about one hour. The following numbers were obtained in the estimation of carbon dioxide in calcite, pure barium carbonate, and pure sodium carbonate :

Calcite.

Weight of Calcite.	Barium Hydroxide Solution.	Hydrochloric Acid required for Titration.	Hydrochloric Acid equivalent to Carbon Dioxide.	Carbon Dioxide found.	Carbon Dioxide calculated.
Grm.	c.c.	c.c.	c.c.	Grm.	Grm.
0·1310	50·0	31·7	32·1	0·0576	0·0575
0·1710	50·0	21·8	42·0	0·0754	0·0751
0·0826	50·0	43·5	20·3	0·0364	0·0363
0·3490	100·0	42·6	85·0	0·1526	0·1534
0·0785	50·0	44·8	19·0	0·0341	0·0345

Hydrochloric acid (approximately decinormal), 1 c.c. = 0·001796 grm. of carbon dioxide.
50·0 c.c. barium hydroxide solution = 63·8 of $\frac{N}{10}$ hydrochloric acid.

Carbon Dioxide in Barium Carbonate (Kahlbaum's).

Weight of Barium Carbonate.	Barium Hydroxide Solution.	Hydrochloric Acid required for Titration.	Hydrochloric Acid equivalent to Carbon Dioxide.	Carbon Dioxide found.	Carbon Dioxide Present calculated.
Grm.	c.c.	c.c.	c.c.	Grm.	Grm.
0·2508	50·0	20·6	32·3	0·0559	0·0558
0·3102	50·0	13·3	39·6	0·0685	0·0691
0·3258	50·0	11·2	41·7	0·0722	0·0726
0·0623	50·0	44·8	8·1	0·0140	0·0138

Hydrochloric acid, 1 c.c. = 0·001732 grm. of carbon dioxide.
Barium hydroxide solution, 50·0 c.c. = 52·9 c.c. of $\frac{N}{10}$ hydrochloric acid.

The apparatus shown in the figure for purifying the carbon dioxide may be simplified if the process described by G. T. Morgan (*Proc. Chem. Soc.*, 1904, 20, 167) is employed. This process consists in using phosphoric acid in place of hydrochloric acid or sulphuric acid to decompose the carbonate; owing to the former acid being non-volatile, it is necessary to remove moisture alone from the carbon dioxide. This can be effected by interposing a single wash-bottle containing concentrated sulphuric acid between the decomposition flask *A* and the apparatus *F*. The following results were obtained :

CARBON DIOXIDE IN SODIUM CARBONATE.

Four Determinations for Each Separate Weight taken.

Sodium Carbonate.	Barium Hydroxide Solution.	Hydrochloric Acid required for Titration.	Hydrochloric Acid equivalent to Carbon Dioxide.	Carbon Dioxide found.	Carbon Dioxide calculated and determined by Titration.
Grm.	c.c.	c.c.	c.c.	Grm.	Grm.
0·1616	50·0	25·5 25·6 25·7 25·7	39·1 (mean)	0·0677	0·0671
0·1081	50·0	38·3 38·4 38·1 38·4	26·4 (mean)	0·0457	0·0448

Hydrochloric acid, 1 c.c. = 0·001782 grm. of carbon dioxide.
Barium hydroxide solution, 50·0 c.c. = 64·7 c.c. of $\frac{N}{10}$ hydrochloric acid.

It is known that greater accuracy may be obtained by using more dilute solutions of barium hydroxide and standard hydrochloric acid. The limit of accuracy is obviously that attainable by Pettenkofer's process—*i.e.*, with ordinary precautions 1 c.c. (0·00196 grm.) of carbon dioxide may be estimated in the gas when the capacity of the flask is about 10 litres.

The method is being used at present for the determination of carbon dioxide in beer, and it is thought it may prove useful for the estimation of carbon dioxide in such materials as cements, clay, soil, soap, and in aerated liquids.

The apparatus is being applied to the determination of small quantities of carbon, methane, and carbon monoxide.

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A QUICK AND ACCURATE METHOD FOR THE ESTIMATION OF BORIC ACID IN MILK AND CREAM.

BY F. W. RICHARDSON, F.I.C., AND WM. KEIGHLEY WALTON.

(*Read at the Meeting, March* 5, 1913.)

THE method of R. T. Thomson when applied to the estimation of boric acid in milk and cream gives results low in proportion to the amount of combustible matter—fat, etc.—in these liquids. The sodium hydroxide does not prevent a serious loss of boric acid when the solids are charred. To three samples of 10 grms. of cream were added respectively 50, 100, and 200 mgrms. of boric acid. By the Thomson process only 21, 60, and 149 mgrms. respectively were found.

We attempted to coagulate milk and cream with mercury, lead, and zinc salts before determining the amounts of boric acid in the filtrate, but with unsatisfactory results. We then tried the following method: Five c.c. of a 5 per cent. solution of sulphate of copper are added to 50 c.c. of milk or 10 grms. cream mixed with 40 c.c. of water. The mixtures are well stirred and are heated to boiling for a few seconds, and the filtrates used for the estimation of boric acid, the contents of the filter having been well washed four or five times with boiling water.

To the cold filtrate 2 c.c. of a 1 per cent. neutralised solution of phenolphthaleïn in alcohol are added and $\frac{N}{10}$ NaOH until a certain blue shade appears. The usual proportion of glycerol, about one-third of the volume, is added, and the titration with $\frac{N}{10}$ NaOH proceeded with until the blue shade again appears. A very little practice enables the right point to be reached. Under these conditions 1 c.c. of $\frac{N}{10}$ NaOH = 0·0071 grm. of boric acid.

Six milks with amounts of boric acid unknown to the analyst gave—

H_3BO_3 Per Cent.						
Present ...	0·045	0·012	0·093	Nil.	0·015	0·006
Found ...	0·041	0·014	0·086	—	0·0124	0·005
Error ...	−0·004	+0·002	−0·007	—	−0·0026	−0·001

Six creams under similar conditions gave—

H_3BO_3 Per Cent.						
Present ...	0·050	0·020	0·010	Nil.	0·150	0·100
Found ...	0·050	0·0202	0·0096	—	0·161	0·1001
Error ...	—	+0·0002	−0·0004	—	+0·011	—

The method is rapid and easy of application, and the results are reasonably accurate. If it is desired to remove the copper, about 0·5 grm. of zinc dust may be added to the boiled mixtures of milk or cream and copper sulphate. The filtrate is colourless. We prefer not to remove the copper, as the small excess of copper acts as an indicator and gives a characteristic blue, as opposed to a green shade when the right amount of $\frac{N}{10}$ NaOH has been added.

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COMBINED ESTIMATION OF BORIC ACID AND FAT IN CREAM.

By F. W. RICHARDSON, F.I.C.

(*Read at the Meeting, March* 5, 1913.)

According to the Public Health (Milk and Cream) Regulations, 1912, it is not permissible to add any preservative substance to cream containing less than 35 per cent. by weight of milk-fat.

This new enactment makes it incumbent upon the analyst to estimate both boric acid and fat in cream. I find that the fat may be easily estimated in the copper coagulum on the filter left in the process above described. The well-drained filter is placed in the mouth of a wide but short-necked and counterpoised flask—a CO_2 flask of about 4 ounces capacity. The flask and filter are kept in the water-oven until the moisture is expelled. The filter is placed in a Soxhlet tube secured into the flask into which most of the milk-fat has filtered. After a brief extraction with petroleum ether, it will be found that the whole of the fat has been recovered. It is necessary to leave the flask containing the fat some three or four hours in the water-oven before weighing; after this period constant weight can easily be obtained. The results are accurate when compared with those given by standard methods.

Discussion.

Mr. T. Macara said in the case of butter he had found as much as 30 per cent. of the boric acid to be lost on ignition with soda. It seemed as though it volatilised in the fat, much in the same manner as it does with methyl alcohol. He had tried the use of caustic lime as well as of caustic soda, because the combustion then was freer; but even this did not seem to fix the boric acid completely. He thought that the Gottlieb method was probably the best for estimating the fat in cream, and by a slight modification the estimation of boric acid could be combined with it.

Mr. Richmond thought that it would be better to titrate at boiling-point. In the method devised by Mr. Miller and himself some years ago, advantage was taken of the fact that, in the presence of a considerable quantity of phenolphthaleïn at boiling temperature, boric acid showed no acidity at all, while when a polyhydric alcohol was added the full acidity was developed, and he thought that if in Mr. Richardson's case the temperature were raised to boiling, the accuracy of the method would probably be improved. The method described for fat estimation appeared to be the old Ritthausen method. It was excellent for cream, though not quite so good for milk, especially when the milk had been heated. As a rapid method he would suggest the estimation of the fat by the Gerber method in a mixture of equal weights of cream and water, and the estimation of the boric acid in a measured quantity of the same mixture by the method devised by Mr. Miller and himself.

Mr. Richardson, in reply, said that they never used a centrifugal method for estimating the fat, being of opinion that the results were not nearly so accurate as were obtained by the method here described. As to the boric acid results, these were certainly on the average a little low, but not to so large an extent as 5 per cent. on the total amount of substance found.

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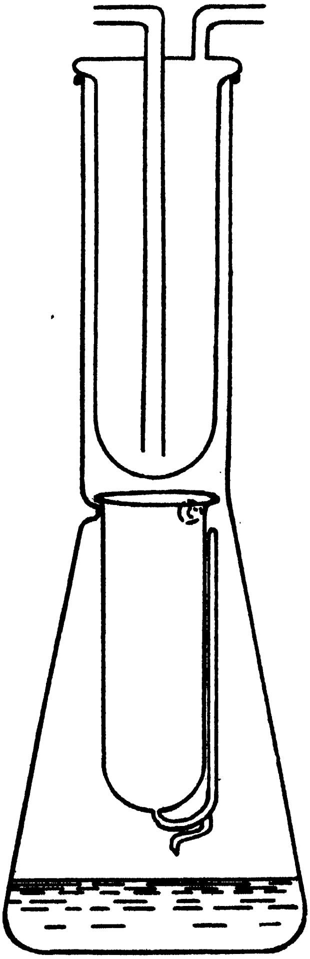
A SIMPLE ALL-GLASS EXTRACTION APPARATUS.

By CLAYTON BEADLE and HENRY P. STEVENS, M.A., Ph.D., F.I.C.

(Read at the Meeting, March 5, 1913.)

In spite of the very large number of extraction apparatus which have been devised, we have always felt the need of a simple Soxhlet apparatus composed entirely of glass without mercury traps, ground-glass joints, or stoppers of any material. There are serious objections to any form of apparatus except one made entirely of glass. Mercury is unhealthy on account of the vapours given off; ground glass joints are very apt to stick, and are expensive; and, of course, rubber or other similar material is unsuitable for use in any part of the apparatus with which the vapours of the solvents come in contact.



The apparatus we are about to describe overcomes these difficulties. It is made entirely of glass, it is efficient, and it is not expensive. It consists essentially of an ordinary Erlenmeyer flask, about 3 inches in diameter at the bottom and 4½ inches in height to the bottom of the neck. The neck is made wide and long, 1⅜ to 1½ inches internal diameter and 4½ inches long. These dimensions have been found convenient, but can be altered to suit circumstances; and the neck could probably be made much shorter if a more efficient and complex condenser were employed. We prefer to use one of the simplest construction, consisting merely of a glass vessel of test-tube shape, provided at the top with a water inlet and outlet, and of about 1¼ to 1⅜ inches external diameter, which is slipped into the neck of the flask and supported by a flange which rests on the rim. The actual extraction-tube consists of a glass thimble with siphon-tube attached, about 3 inches long and 1 to 1¼ inches external diameter. The thimble hangs by means of a flange on three or four projections at the bottom of the neck of the flask, made by softening the glass and forcing a sharp-pointed instrument in to a depth of about ¼ inch. The method of working is self-explanatory. It is found that the condenser is sufficient for the complete condensation of the vapours of such volatile solvents as ether and acetone. We have tested the apparatus constantly in practice, and find no loss after extraction for eight hours. The apparatus has the advantage that the substance to be extracted is immersed in the solvent or its vapour at the boiling-point of the liquid. It is therefore more efficiently extracted than in the ordinary siphon Soxhlet apparatus, where the liquid in the extraction-tube is cooled by radiation.

In order to facilitate the removal of the extraction-thimble a piece of fine metal wire may be twisted round it just below the flange, leaving two ends on opposite sides 2 or 3 inches long. When the tube is inserted, these will lie against the walls of the neck within easy reach of the fingers, so that by pulling the wires the thimble may be easily withdrawn.

The only possible objection which can be raised against this apparatus is the fact that it is more convenient to transfer the contents to a separate flask for the evaporation of the solvent and the weighing of the extracted material. We do not ourselves see any objection to doing this, but, as pointed out, the neck and condenser could probably be shortened, and the total height of flask and neck reduced to about 7 to 8 inches, when it would not be too high to go on to the pan of the ordinary balance.

We have now had this apparatus in constant use for some months in our laboratories, particularly for the extraction of rubber and cellulose products, and have found it satisfactory. A simple and light form of clip is all that is necessary to support it in an upright position when standing on a water-bath or wire gauze.

The apparatus may be obtained from Messrs. Baird and Tatlock.

DISCUSSION.

Mr. R. ROSS observed that an apparatus of this kind would be very useful for the analysis of samples of spent oxide in gasworks, where a number of determinations had to be made at one time.

Mr. E. R. BOLTON remarked that the apparatus might be further simplified, if desired, by placing some cotton wool at the bottom of the extraction tube and a disc of filter-paper on the top, thus doing away with the siphon. In the ordinary Soxhlet apparatus the solvent easily became cold, and, moreover, sometimes some of the solvent was retained in the centre by capillarity, and never passed round.

Dr. STEVENS said that they should like to try the apparatus without the siphon tube before expressing an opinion as to the desirability of doing away with this. A good deal of evaporation went on, and it was not easy to say how much of the liquid would drop through; some might possibly evaporate again. Used as a siphon apparatus, however, in the ordinary way, the apparatus worked perfectly well.

Mr. BEADLE said that when the apparatus was working, the solvent could be seen boiling in the syphon thimble during the whole time, while it was surrounded by its own vapour. A number of pieces of apparatus could be connected together by rubber tubing, and placed upon a thin iron plate heated by small burners, without any water-bath, and any single flask could be removed without disturbing the others. The contents of the flask must, of course, be transferred to another flask for evaporation of solvent and weighing, but this entailed very little inconvenience, and, indeed, had certain advantages.

In reply to a question put by the PRESIDENT, Mr. Beadle said that, although the solvent in the thimble could be seen to be gently boiling, this did not interfere with the proper siphoning of the liquid.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Copper in Cocoa and Chocolate. C. Formenti. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1913, **25**, 149-154.)—Three samples of cocoa beans (after removal of the shell) yielded from 0·020 to 0·034 grm. of copper per kilo. of substance, whilst the shells separated from the beans were found to contain 0·014 to 0·040 grm. of copper per kilo. These results agree with those found by most other observers, although Gautier (in a book published in 1883) has stated that cocoa-shell contains 0·225 grm. of copper per kilo. Various samples of chocolate examined contained quantities of copper varying with the amount of cocoa present and in proportion to the above-mentioned quantities. W. P. S.

Estimation of Coumarin in Melilotus. E. Obermayer. (*Zeitsch. anal. Chem.*, 1913, **52**, 172-191.)—The method consists essentially in extracting the coumarin with ether, evaporating the extract, and distilling it from calcium chloride solution, and oxidising the distillate with permanganate. Ten grms. of the powdered, air-dry sample are extracted with ether, and the ethereal extract is evaporated in a 500-c.c. flask until the ether has been removed completely. Three hundred c.c. of calcium chloride solution (1 kilo. of the dry salt to 3 litres of water) are then added, and the mixture is distilled as far as possible, a small quantity of pumice-stone being added to the flask to prevent "bumping." The distillate is diluted to 500 c.c., filtered, and a quantity of the filtrate corresponding with from 0·005 to 0·05 grm. of coumarin is treated with from 25 to 50 c.c. of pure 20 per cent. zinc sulphate solution. After the addition of from 25 to 50 c.c. of $\frac{N}{10}$ potassium permanganate solution, the mixture is boiled for ten minutes, then filtered through an asbestos filter, the insoluble residue washed with water, and the excess of permanganate in the filtrate titrated with $\frac{N}{10}$ oxalic acid solution after the addition of sulphuric acid. Each 0·01 grm. of coumarin requires 43·365 c.c. of $\frac{N}{10}$ permanganate for oxidation. When applied to pure coumarin, the process yields only slightly over 95 per cent. of the quantity present, and the amount found must therefore be multiplied by 1·04835 to obtain the actual quantity of coumarin in the sample. The process may be shortened by acidifying the oxidised and cooled solution and titrating the excess of permanganate without an intermediate filtration, but the results obtained are not as trustworthy as those given by the method first described. W. P. S.

Acid-Butyrometric Method for the Estimation of Fat in Cheese and Milk Products. Hammerschmidt. (*Milchwirtschaftl. Zentralbl.*, 1912, **41**, 757-763; through *Chem. Zentralbl.*, 1913, I., 467.)—The butyrometer tube employed is open at both ends, and the graduated portion shows up to 35 per cent. of fat in 0·5 per cent. divisions. Two and a half grms. of cheese are placed in the tube, together with 9 c.c. of "solvent" (*cf.* O. Wendler below) and 1 c.c. of amyl alcohol, and the mixture is heated to 40° C. and shaken until the casein has dissolved. The contents of the tube are then cooled until the fat begins to solidify, when 10 c.c. of sulphuric acid are added, the mixture is shaken thoroughly, and submitted to centrifugal action. W. P. S.

Estimation of Fat in Cheese by the Acid-Butyrometric Method without the Use of Amyl Alcohol. W. L. Kooper. (*Milchwirtschaftl. Zentralbl.*, 1912, **41**, 753-757; through *Chem. Zentralbl.*, 1913, I., 466-467.)—Two and a half grms. of the cheese are placed in a butyrometer tube (a form of tube open at both ends is employed; see following abstract), the wide end of the tube is closed with a rubber stopper, and 6·5 c.c. of sulphuric acid of sp. gr. 1·54 are introduced through the other end of the tube. The cheese is then dissolved by immersing the tube in hot water and shaking the contents occasionally. When the casein has dissolved, 6·5 c.c. of sulphuric acid of sp. gr. 1·82 are added, and the mixture is submitted to centrifugal action. W. P. S.

Estimation of Fat in Cheese by the "Neusal" Method. O. Wendler. (*Milchwirtschaftl. Zentralbl.*, 1912, **41**, 763-765; through *Chem. Zentralbl.*, 1913, I., 467.)—A new form of butyrometer tube for use in this method is described (*cf.* Golding, ANALYST, 1911, **36**, 203). The graduations on the tube enable the volume of the separated fat to be read within 0·1 per cent. The casein solvent used consists of a solution containing salicylates and thiosulphates, and the fat-clarifying reagent consists of butyl alcohol. For each test 2·5 grms. of cheese, 12 c.c. of solvent, and 1 c.c. of butyl alcohol are employed; solution of the casein is attained by heating the mixture in a water-bath, and the volume of the fat is read off at 70° C.

W. P. S.

Estimation of Water in Cheese. C. Mai and E. Rheinberger. (*Study Committee Internat. Dairy Federation: Proposal for the Unification of Analytical Methods for Cheese*, No. 1913.) The method devised by Hoffmann (*Zeitsch. angew. Chem.*, 1902, **15**, 1193), and subsequently recommended with various modifications by others (ANALYST, 1907, **32**, 21; 1908, **33**, 97; 1911, **36**, 586), has been tested upon cheese. Of the immiscible solvents, toluene, xylene, turpentine oil, and petroleum oil, only the last was found suitable. Special experiments proved that the water distilled quantitatively with the petroleum oil, and that none was lost through emulsification; but it was found essential to collect the condensed water in a receiver, the outlet of which was closed by means of a second (reflux) condenser. From 8 to 12 grms. of the cheese are placed in a 300 c.c. Erlenmeyer flask with a mark at 200 c.c. Petroleum oil is added up to this mark, a few fragments of pumice introduced, and the flask connected with the condenser and heated on a sand-bath over a fairly small flame until the graduated receiver contains about 75 c.c., and the lower aqueous layer no longer increases (thirty to forty-five minutes). The distillation is then stopped, and the receiver disconnected and immersed in water at 15° C. for thirty minutes, after which the volume of the aqueous layer is read, and calculated into the percentage in the sample. As a rule, though not invariably, the method gives slightly higher results than those calculated from the loss on drying, but the results of duplicate estimations agree well. Special precautions must be taken to obtain average samples for the estimation. C. A. M.

Rapid and Accurate Gravimetric Method for determining Milk-Fat in Evaporated Milk and Milk Powders. E. P. Harding and G. Parkin. (*J. Ind. and Eng. Chem.*, 1913, **5**, 131-134.)—The method is a modification of one devised by L. Nye and one of the authors for determining fat in cereals, the principle being the breaking-down of the milk emulsion by dissolving all the proteins in acetic acid (25 per cent. by volume), the solution of the fat in a mixture of alcohol and carbon tetrachloride, and its extraction with petroleum ether (b.-pt. between 50° and 70° C.). With evaporated milks either the material itself or a 40 per cent. emulsion can be used, the latter giving less chance of error in sampling. Forty grms. of the well-mixed sample are transferred to a flask, diluted to 100 c.c., well shaken, and 4 c.c. pipetted into a Nessler jar, or a quantity equivalent to 1·6 grms. of the original sample is weighed out. Eight c.c. of the 25 per cent. acetic acid are added, and the whole is warmed to about 50° C. As soon as the proteins are dissolved 12·5 c.c. of carbon tetrachloride are added, and the flask is stoppered and vigorously shaken for two minutes, after which 25 c.c. of alcohol are added and the mixture again well shaken. Twenty-five c.c. of petroleum ether are next added, and after more shaking another 15 c.c., followed by further shaking. The flask is now allowed to stand for two minutes for the layers to separate; if, as rarely happens, an emulsion is formed, a few drops of alcohol will cause it to break. The upper layer is blown off by means of a wash-bottle fitting and filtered into a tared weighing-bottle, and three successive washings of ether and carbon tetrachloride are also blown off in the same manner. The fat is dried for an hour at 100° C. and weighed. On an average 0·4 per cent. more fat is obtained from a milk containing about 8 per cent. than by the Röse-Gottlieb method (as described in Circular 66 *Dept. of Agric.*), and 0·16 per cent. more than is obtained by using Hortvet's modification of the Röse-Gottlieb method, in which one-half the amount of sample is used and one additional blow-off is made. Good results are also obtained in the analysis of ice-cream.

H. F. E. H.

Oil from the Seeds of "Ximenia Americana." F. Schröder. (*Arbeiten aus dem Kaiserl. Gesundheitsamte*, 1912, **43**, 454-474.)—*Ximenia Americana, L.*, a tree belonging to the *Olacaceæ* is indigenous to tropical parts of America, Asia, and Africa, and is met with in the wild state in German East Africa, where its fruit is eaten by the natives. The seeds of the fruit contain kernels which, in the case of the samples examined, yielded from 65·05 to 67·34 per cent. of a light yellow oil. No hydrocyanic acid was present, but about 1 per cent. of a rubber-like substance could be precipitated with acetone from the ethereal extracts of the oil. To this body was attributed the remarkably viscid character of the oil. The oil became partially solid at 8° C., and completely solid at - 4° C. It gave the following values:

	Oil Expressed.	Oil Extracted with Ether.	Oil Extracted with Acetone.
Specific gravity at 15° C.	0·9205	0·9220	0·9210
Acid value	10·5	11·76	12·0
Saponification value	173·6	173·2	177·0
Iodine value	81·3	85·05	80·3
Hehner value	94·8	93·9	94·8
Polenske value	0·14	0·12	0·21
Unsaponifiable matter, per cent. ...	0·46	0·55	0·46
Refractive index at 40° C. (Abbé) ...	1·4680	1·4687	1·4677
Viscosity at 50° C. (Engler) ...	8·5	11·3	6·6
Neutralisation value of fatty acids ...	180·7	180·2	184·4
Mean molecular weight of fatty acids...	310·8	304·6	304·6

The fatty acids melted at 49·5° C. to 50° C., solidified at 44·4° C., and had an iodine value of 71·01. They consisted of about 75 per cent. of liquid acids, and 25 per cent. of solid acids, including about 10 per cent. of arachidic acid. The oil had a pungent taste, and was unsuitable for food. C. A. M.

Estimation of Soluble Nitrogenous Substances as a Criterion of the Quality of Flour. E. Rousseaux and M. Sirot. (*Ann. Falsificat.*, 1913, **6**, 78-84.)—In flour derived from fully-ripened grain, the nitrogenous substances are chiefly in an insoluble condition, whilst in that from unripe grain there will be a much larger proportion of soluble nitrogenous substances. Hence the ratio between the total nitrogen and the soluble nitrogen affords a criterion of the condition of the flour. The soluble nitrogen is estimated by shaking 10 grms. of the flour with about 150 c.c. of water until thoroughly mixed, and then placing the flask for two to five minutes on the boiling water-bath, with frequent agitation. Its contents are next cooled, made up to 200 c.c., thoroughly shaken and filtered, and the nitrogen estimated in 50 c.c. of the filtrate. The ratio between the total nitrogen and soluble nitrogen ranged from 5·30 : 1 to 6·02 : 1 in the case of twenty typical samples of good flour of all grades, the average being 5·72 : 1. In the case of seven samples of inferior flour, the ratio varied from 1·15 : 1 to 2·60 : 1. Flour kept under bad conditions also showed a low ratio. Thus, a flour with a ratio of 5·69 : 1 was kept for three months under conditions that promoted germination, and then showed a ratio of 3·78 : 1. Should the ratio be less than 5·20 : 1, as in the case of old flour, it will be found in practice that difficulties will be experienced in baking bread from that flour.

C. A. M.

Estimation of Glyceryl Acetate in Essential Oils. S. G. Hall and A. J. Harvey. (*J. Soc. Chem. Ind.*, 1913, **32**, 61-62.)—Schimmel's method is reported on favourably by Salaman and Seaber (*J. Soc. Chem. Ind.*, 1912, **31**, 1052), and depends upon the solubility of glyceryl acetate in 5 per cent. alcohol and its consequent elimination from the oil on washing, 2 per cent of glyceryl acetate being detectable. The authors consider a better method is the following, which depends upon the separation and estimation of the glycerol as such. Not less than 10 grms. of the oil to be examined are mixed with about 50 c.c. of alcohol (sp. gr. 0·830), and saponified with $\frac{N}{2}$ alcoholic potash; the solution is then digested on the water-bath for an hour, neutralised with $\frac{N}{2}$ hydrochloric acid, and evaporated to dryness in the water-bath to remove the alcohol. About 20 c.c. of water are added, and the oily portion extracted with ether, the aqueous solution being run into a flask. The ether extract is again washed with about 10 c.c. of water, which is then added to that already in the flask, and the whole evaporated to a syrup, which now contains the glycerol originally present as glyceryl acetate. This can be estimated in the usual way by the triacetin process; 1 c.c. $\frac{N}{2}$ caustic soda $=$ 0·01535 glycerol, or 0·03641 glyceryl acetate. Results obtained with Bergamot and lavender oils are appended. H. F. E. H.

Composition of Russian Honey. E. J. Sarin. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1913, **25**, 131-139.)—Results of analyses of seventy-two samples of Russian honey are reported, the data obtained being as follows:

	Minimum.	Maximum.	Average.
	Per Cent.	Per Cent.	Per Cent.
Water	14·80	21·79	16·39
Sucrose	0	5·49	1·90
Invert sugar	65·64	79·20	74·91
Ash	0·03	1·02	0·18
Non-sugars	2·20	14·98	5·96
Acidity (as formic acid)	0·03	0·20	0·11
Proteins	0·10	1·56	0·44
Tannin precipitate (Lund's test)	0·5 c.c.	7·5 c.c.	2·0 c.c.
Polarisation 10 per cent. solution:			
Before inversion	−0·81°	+3·82°	—
After inversion	−0·82°	+3·02°	—

The samples yielded negative reactions with Ley's, Fiehe's, Jagerschmid's, Brown's, and Armani-Barboni's tests, except in the case of three samples which gave a positive reaction with Ley's test. Five of the samples examined were obtained from bees which had been fed with sugar; these samples could not be distinguished from the others. W. P. S.

Estimation of Iron in "Extractum Ferri Pomati." K. Kropat. (*Arch. Pharm.*, 1913, **251**, 90-93.)—One grm. of the extract is weighed in a stoppered Erlenmeyer flask and gently heated with 30 c.c. of dilute sulphuric acid until dissolved. After cooling, an addition of 1 grm. of very finely-powdered potassium permanganate is made, and the flask shaken for a couple of minutes and heated for two to three minutes on the steam-bath, after which it is allowed to cool, with frequent shaking, until manganese peroxide particles can no longer be seen in the light yellow solution. The sides of the flask are then washed down with not more than

10 c.c. of water, 2 grms. of potassium iodide introduced, and the flask tightly stoppered and allowed to stand for an hour, after which its contents are diluted with 100 c.c. of water and titrated with $\frac{N}{10}$ sodium thiosulphate. At least 9 c.c. (= 5 per cent. of iron) should be required. C. A. M.

Estimation of Morphine in Opium and Opium Preparations. G. Guérin. (*J. Pharm. Chim.*, 1913, 7, 162-163.)—*Opium.*—A quantity of 7·5 grms. of the powdered opium, dried previously at 60° C., is mixed with 3 grms. of calcium hydroxide and 30 c.c. of water in a mortar; the mixture is then transferred to a 125 c.c. flask, and the mortar and pestle are rinsed with water, using 45 c.c. of the latter. The contents of the flask are shaken gently at intervals during two hours and then filtered. Fifty-two c.c. of the filtrate are placed in a flask, 5 c.c. of pure acetone are added, and 1 grm. of ammonium chloride is dissolved in the mixture. After the lapse of twenty-four hours, the crystals which form are collected on a counterpoised filter, washed with water saturated with morphine until the washwater is colourless and free from chlorides, and then four times with acetone saturated with morphine, 15 c.c. of acetone being used each time. The filter and its contents are dried at 100° C. and weighed.

Extract of Opium.—Three grms. of the extract are dissolved in 75 c.c. of water, the solution is treated with 3 grms. of calcium hydroxide, and, after the lapse of three hours, the mixture is filtered. Sixty-five grms. of the filtrate (equivalent to 2·5 grms. of extract) are then subjected to the process described above.

Tincture of Opium.—Seventy-five grms. of the tincture are evaporated to dryness, the residue is treated with 75 c.c. of water, and the liquid is mixed with 3 grms. of calcium hydroxide. The mixture is shaken for two hours, then filtered, and the morphine is estimated as described in 52 c.c. (50 grms. of tincture) of the filtrate. W. P. S.

Analysis of Peru Balsam. H. R. Jensen. (*Pharm. J.*, 1913, **90**, 210-211.)—Artificial products closely resembling Peru balsam are now manufactured; they consist of a perfumed mixture of synthetic esters with resinous and fatty matters, and, on analysis, yield results which are generally within the range of figures given by genuine balsams. It was found, however, that the cinnamein extracted from the artificial balsam differed from that of the natural balsam as regards its iodine value and optical properties. The iodine value of the artificial cinnamein was 1·5, and the refractive index at 15° C., 1·565; the cinnamein from natural balsam had an iodine value of 25·5 and a refractive index of 1·575. Further difference was noticed when the cinnameins were fractionally distilled under reduced pressure (15 mm.). The first fraction (10 per cent. of the total) of the natural cinnamein had a sp. gr. of 1·044, a refractive index of 1·5355, and a specific rotary power of +5°, whilst the artificial cinnamein (first fraction) had a sp. gr. of 1·09, a refractive index of 1·555, and was optically inactive. The fourth fraction of the natural cinnamein distillate still exhibited a slight optical rotation. W. P. S.

Detection of Saponin. L. Rosenthaler. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1913, **25**, 154-158.)—The test proposed by C. Sormani (*cf.* ANALYST, 1912, **37**, 350) fails occasionally owing to the fact that certain saponins are devoid of hæmolytic action, and the author therefore describes a test which is stated to be capable of giving a positive reaction with saponins derived from any source. The test depends not on the separation of the saponin, but on reactions of the product obtained from it by hydrolysis. The product is not the substance produced by theoretically complete hydrolysis—namely, sapogenin or endosapogenin—but an intermediate product, in which the sapogenin is still combined with a quantity of sugar; this intermediate product is termed prosapogenin. It yields an orange-red coloration with concentrated sulphuric acid, the colour changing slowly to red, and then (after a few hours) to violet. When shaken with sodium carbonate solution prosapogenin yields a persistent froth. The liquid to be tested for the presence of saponin is treated with hydrochloric acid, so that about 2·5 per cent. of the acid is present in the mixture, and the latter is then heated on a water-bath until hydrolysis is apparently complete—that is, until the mixture ceases to give a froth when shaken. If a precipitate forms when the acid is added, the mixture must be filtered before hydrolysis. The mixture, while still warm, is shaken with about one-half its volume of ethyl acetate, the ethyl acetate is separated, washed with small quantities of water until free from hydrochloric acid, decolorised with animal charcoal, and evaporated. The residue is then tested as described. In the case of beer, the sample is heated with an equal volume of 95 per cent. alcohol until a flocculent precipitate forms, filtered, and the filtrate submitted to hydrolysis after the alcohol has been removed. Or, the beer may be evaporated, the residue extracted with hot 70 per cent. alcohol, and the filtered alcoholic extract then hydrolysed and tested. W. P. S.

Detection and Differentiation of Poisonous and Non-Poisonous Saponins by Hæmolysis. J. Halberkahn. (*Deutsch. Mineralwasserfabrikanten-Zeit.*, 1912, Nos. 25-30; through *Chem. Zentralbl.*, 1913, I., 852.)—The use of saponins in the manufacture of mineral waters should be restricted to those which have been deprived of their poisonous constituents. This may be effected by means of cholesterin, treatment with hot barium hydroxide solution, or saponification of the acetyl compound with barium hydroxide or caustic alkalis. The toxicity of saponins can be determined, and poisonous saponins distinguished from non-poisonous, by hæmolysis. For the isolation of saponins the method of Brunner as elaborated by Rühle (ANALYST, 1908, **33**, 408) is alone valid. Concentration of mineral waters without previous neutralisation must be avoided, and the author prefers to eliminate heating, although it may be justified in the case of liquids containing dextrin (Brunner and Rühle, *loc. cit.*), which must, however, be neutral during concentration. The extraction with phenol is carried out in two stages, with 10 and with 5 c.c. The aqueous liquid which always accompanies the phenol extract is removed either by treating with 15 c.c. of absolute alcohol and drying with freshly-ignited sodium sulphate, or by means of a centrifugal; the extract is then treated with water and ether in a separating funnel, into which it is rinsed with a mixture of equal volumes of phenol and absolute alcohol. The aqueous layer is evaporated at 60° C., and dried in a vacuum desiccator without heating. A disadvantage of the method is that water-soluble coal-tar colours find their way into the phenol extract, and interfere, particularly the red ones, with the hæmolytic test. Animal charcoal cannot be used, as it removes the saponins also. Poisonous and non-poisonous saponins show much the same behaviour to hæmolysis, whether extracted by the Brunner method from mineral waters, or obtained from other sources, so that this method is without effect on them. Solutions from saponin deprived of its poisonous constituents and solutions containing no saponin give residues showing no hæmolysis. Glycyrrhizin has no hæmolytic action, and cannot be confused with poisonous saponins. O. E. M.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Occurrence of Formaldehyde in the Juices of Plants. F. Angelico and G. Catalano. (*Gazz. Chim. Ital.*, 1913, **43**, 38-43.)—The plant *Atractylis gummifera* contains an active glucosidal principle, *atractiline*, which gives a specific reaction with formaldehyde, and will detect as little as 3 drops of 40 per cent. formalin solution in a litre of water. By means of this reagent the presence of formaldehyde in the juices of the green leaves of numerous plants has been established, the results being checked by tests applied to the distillates from the juices. On treating the glucoside, atractiline, with 2 or 3 drops of concentrated sulphuric acid, a yellow coloration is produced at the point of contact, and on now adding a drop of a liquid containing a trace of formaldehyde a violet coloration, appearing bright blue in reflected light, is produced. This reaction is not obtained with other aldehydes. The formation of formaldehyde in the cells of the green plant appears to be connected with the phenomena of the chlorophyll function. Thus formaldehyde was found in the juices of the leaves of eleven plants (including *Lupinus albus*, *L. gorgonia*, *Zea maïs*, etc.) that had been exposed normally to the light during their growth, but it was absent from the juices of the leaves of the same plants after being kept for twenty-four hours in the dark. No formaldehyde was found in the juices or distillates of several species of fungi, such as *Psalliota campestris*. C. A. M.

Respiratory Quotients of Green Plants. L. Maquenne and E. Demoussy. (*Comptes rend.*, 1913, **156**, 278-283.)—The authors find that the respiratory quotient of plants (see ANALYST, 1913, 108) always exceeds unity when the plants are examined in the spring or early summer. Out of some thirty-eight different plants investigated, *Aspidistra* was the only exception to this rule, the respiratory quotient being 0·94. Later in the season (autumn) the value of the quotient decreases, and it may be concluded that the respiratory quotient of green leaves is greater than unity during their period of active vegetation, and that degeneracy is indicated when the quotient is less than unity. It was also found that the percentage quantity of nitrogen in the atmosphere increased slightly in cases where the respiratory quotient was low, whilst it diminished when the plants yielded quotients above unity.

W. P. S.

Detection of Adulteration by the Serum Precipitation Method. A. Vila. (*Ann. Falsificat.*, 1913, **6**, 84-89.)—A method is described of obtaining specific anti-sera by treating rabbits with the blood-plasma instead of the serum from the animal in question. By the use of the rabbit serum thus prepared it is possible to detect with certainty the presence of at least 5 per cent. of, *e.g.*, horseflesh in sausages.

C. A. M.

Measurement of Tryptic Protein Hydrolysis by Determination of the Tyrosine Liberated. S. J. M. Auld and T. D. Mosscrop. (*J. Chem. Soc.*, 1913, **103**, 281-284.)—The method employed is based, with modifications, on that described by Brown, Millar, and others (*Trans. Guiness Research Lab.*, 1903, Part I.), and consists of the estimation by absorption of bromine of the tyrosine formed during tryptic digestion. As described (*loc. cit.*), the end-point of the reaction was indicated by the persistence of the yellow colour of bromine, but the author points out that in ordinary protein digests yielding coloured solutions this is impossible; nor is the use of starch and potassium iodide permissible, since hydrochloric acid will of itself liberate sufficient iodine from the iodide to colour starch.

It was found that good results were obtained when methyl violet or gentian violet were used, as outside indicators dissolved in 70 per cent. alcohol, at the rate of 1 per cent. About 10 drops of this solution are added to 10 c.c. of 5 per cent. hydrochloric acid, and dotted over a white tile. The addition of sodium bromate or traces of free bromine causes a change from olive-green (the colour of the dye-stuff in acid solution) to a deep bluish-violet. The change is sharp, but the colour fades after a few minutes. Dibromotyrosine only is apparently formed, and the absorption at first is very rapid, but falls off considerably towards the end of the reaction, as experiments cited show. The solution to be titrated is made of not more than 5 per cent. acidity with hydrochloric acid; a lower concentration than 2 per cent. turns the indicator blue, while more than 5 per cent. turns it yellow. To the acid solution is added 15 to 20 c.c. of 20 per cent. sodium bromide, and the liquid is then titrated with $\frac{N}{20}$ sodium bromate. Towards the end of the reaction at least thirty seconds should elapse between successive additions of the bromate, and the solution should be well shaken in a stoppered bottle.

Results quoted show a plus error varying from 0·4 to 2·3 per cent. In two of the test liquids there were present, in addition to tyrosine, leucine, asparagin, and ammonium chloride. Experiments carried out with edestin digested in dilute sodium carbonate solution with trypsin, and corrected for the bromine absorbed by the protein, confirmed Brown and Millar's (*J. Chem. Soc.*, 1906, **89**, 145) contention that the method can be used for determining tyrosine in presence of proteins and other early cleavage products, and also that practically all the tyrosine is liberated in the first stages of tryptic digestion.

H. F. E. H.

Method for the Estimation of Mercury in Urine and Animal Tissues. H. Buchtala. (*Zeitsch. physiol. Chem.*, 1913, **83**, 249-303.)—The mercury is separated electrolytically from the liquid obtained after the destruction of the organic matter by treatment with hydrochloric acid and potassium chlorate. In the case of urine, 1 litre of the sample is heated with 10 per cent. of its volume of concentrated hydrochloric acid, and small quantities of potassium chlorate are added from time to time until the mixture on evaporation yields a light yellow liquid measuring a few c.c. This residual liquid is diluted, filtered, and transferred to a beaker of about 400 c.c. capacity; a porous cell filled with dilute ammonium oxalate solution is placed in the solution in the beaker, and the anode (a carbon rod) is immersed in the ammonium oxalate solution. The cathode consists of a piece of gold foil, 2·5 mm. by 3·5 mm., and 0·015 mm. in thickness, and is suspended in the solution contained in the beaker by means of a platinum wire. Using a current of 1 ampere and 5 volts, the mercury which may be present will be deposited completely on the cathode in from four to six hours. The cathode is now removed, washed with dilute hydrochloric acid to remove traces of iron, then with water, alcohol, and ether, dried in the air, and weighed. The mercury may be volatilised from the gold foil by heating the latter in a tube, and the sublimate obtained can be identified as mercury by treatment with iodine vapour. Experimental proof is given that the method is trustworthy. The author also gives the results of an investigation as to the rate, etc., of the elimination of mercury from the body after courses of treatments with mercury compounds, and appends a comprehensive list of the literature dealing with the subject. W. P. S.

Nitroprusside Reaction of Urine. V. Arnold. (*Zeitsch. physiol. Chem.*, 1913, **83**, 304-314.)—The colour reaction described previously by the author (ANALYST, 1907, **32**, 94) is yielded by the urine of persons in normal health who have recently consumed food—especially flesh foods—but not of persons suffering from high fever. W. P. S.

Colorimetric Method for the Estimation of Uric Acid in Urine. O. Folin and A. B. Macallum, jun. (*J. biol. Chem.*, 1913, **13**, 363; through *Chem. Zentralbl.*, 1913, I., 852.)—The residue obtained by evaporating 2 to 5 c.c. of urine with 1 drop of saturated oxalic acid solution is twice extracted for five minutes in the cold with 10 to 15 c.c. of a mixture of 2 parts ether and 1 part methyl alcohol, and dissolved in 5 to 10 c.c. of water containing 1 drop of saturated sodium carbonate solution. To the solution is added 2 c.c. of the uric acid reagent of Folin and Macallum (ANALYST, 1912, **37**, 355) and 20 c.c. saturated sodium carbonate solution, and the blue liquid resulting is diluted to 100 c.c. in a measuring flask. It is then compared with a standard solution containing 0·001 grm. uric acid, lithium carbonate, 2 c.c. of the uric acid reagent, and 20 c.c. of sodium carbonate solution. O. E. M.

ORGANIC ANALYSIS.

Quantitative Study of Some Aldehyde Reactions. B. G. Feinberg. (*Amer. Chem. J.*, 1913, **49**, 87-116.)—Methods of estimation based on the combination of aldehydes with bisulphite and titration of the excess of the latter with iodine suffer from the drawback that the compound produced is readily broken up again into its components by dilute acids and alkalis. The end-point of the iodine titration is uncertain, owing to the continuous liberation of fresh bisulphite from this cause; the instability of the compounds is increased by the presence of a hydroxyl group in proximity to the aldehyde group. Formaldehyde may be titrated in this way with an accuracy of 98 to 99 per cent. Approximate results (93 to 95 per cent.) may be obtained with vanillin and benzaldehyde, but with salicylic, *p*-hydroxybenzoic and anisic aldehydes the method is useless. A 1 per cent. solution of the aldehyde is treated with 10 to 25 c.c. of a 3 per cent. solution of sodium bisulphite, and the excess of the latter is titrated back with iodine, after 15 to 25 minutes, until the blue colour with starch persists for an instant. The best results are obtained with an excess of bisulphite equivalent to 25 to 50 c.c. of $\frac{N}{10}$ iodine. In the methods based on the use of neutral sulphite, the latter is supposed to combine with the aldehyde, liberating sodium hydroxide. This did not occur in the cases studied, but a reaction took place on the addition of acid, owing to the formation of bisulphite. With benzaldehyde, this method may be used, giving satisfactory results (98 to 99 per cent.), provided a fairly large quantity of aldehyde be taken. Example: About 1 grm. of benzaldehyde is weighed out into a stoppered flask with 10 c.c. of alcohol and about 10 c.c. of water, to wash down the sides of the flask. Six drops of a 1 per cent. solution of phenolphthaleïn are added, and the solution is neutralised with $\frac{N}{10}$ alkali. Then 50 c.c. of a 20 per cent. solution of sodium sulphite, neutralised to phenolphthaleïn, are added, and the mixture is titrated not too rapidly with $\frac{N}{2}$ sulphuric acid. The titration is controlled by a blank containing 35 c.c. of the sulphite solution (*i.e.*, the amount calculated to be left unabsorbed), and the quantity of acid required to neutralise this is deducted from the acid used in the main titration. The same method applied to formaldehyde, but without the use of alcohol, also gave satisfactory results. Anisic aldehyde presented some difficulty about the end-point, more phenolphthaleïn having to be added during the titration, and the results were low (94 per cent.). Salicylic aldehyde, *p*-hydroxybenzaldehyde, and vanillin gave coloured solutions which could not be titrated.

Among the precipitating reagents, *p*-bromphenylhydrazine gives good results with *p*-hydroxybenzaldehyde, vanillin, and anisic aldehyde: Twenty-five c.c. of a 1 per cent. solution of the aldehyde in water, aqueous alcohol, or aqueous acetic acid are mixed with 75 c.c. of a solution of the hydrazine in hot water, equivalent to two to three times the amount theoretically required. Precipitation is effected at 50° C. and the precipitate allowed to stand for one to five hours, collected in a Gooch crucible, washed with hot water, and dried at 105° C. This reagent is not satisfactory for formaldehyde, benzaldehyde, and salicylic aldehyde. On the other hand, *p*-nitrophenylhydrazine is a good general precipitating reagent for aldehydes. It is particularly suitable for the estimation of vanillin and *p*-hydroxybenzaldehyde; with benzaldehyde, salicylic, and anisic aldehydes the results are subject to errors of 2 to 4 per cent. For benzaldehyde, 25 c.c. of a 1 per cent. solution of the aldehyde in 12 per cent. acetic acid are diluted with 50 c.c. of water and 30 c.c. of 30 per cent. acetic acid containing about double the theoretical quantity of *p*-nitrophenylhydrazine are added. The precipitate is allowed to stand for five hours, collected in a Gooch crucible, washed with 10 per cent. acetic acid, and dried at 105° C. For salicylic aldehyde and anisic aldehyde the conditions are essentially the same. For vanillin and *p*-hydroxybenzaldehyde, it is best to use a solution of *p*-nitrophenylhydrazine in $\frac{N}{1}$ hydrochloric acid, adding this drop by drop to the aldehyde solution previously diluted with 75 c.c. of water, stirring the liquid all the time, and allowing the precipitate to stand for half an hour before filtration. With formaldehyde the precipitation method gives results much too low, and volumetric methods are to be preferred. J. F. B.

Quantitative Estimation of Natural in Presence of Artificial Asphaltum. J. Marcusson. (*Zeitsch. angew. Chem.*, 1913, **26**, 91-93.)—The detection of natural asphaltum in the presence of coal-tar pitch is commonly based on the higher proportions of sulphur and mineral matters generally present in the natural products. Such methods, however, are open to the objection that the proportions of these constituents may readily be increased artificially to lead to erroneous conclusions. The method now described is based on a constitutional difference between the bituminous portions of the two types of material, in that the bitumens of coal-tar pitch, being members of the aromatic group, readily yield sulphonic acids soluble in water, whereas the bitumens of all varieties of natural asphaltum are converted by sulphuric acid into insoluble carbon-like derivatives, richer in sulphur than the original bitumen. With pure Trinidad asphaltum the product of the reaction with sulphuric acid shows a gain in weight of about 8 per cent., and the bitumens of Syrian and Mexican asphaltums show a similar increase; the aqueous extracts of these products are nearly colourless, whereas those from the sulphonation of coal-tar pitch are highly coloured. The author has made experiments to see how far this reaction is valid as a means for quantitative estimation in mixtures, but has found that the accuracy is considerably impaired by the fact that, in presence of coal-tar pitch, the bitumens of the natural asphaltums do not give a quantitative yield of insoluble sulphonation products, so that a substantial correction must be applied to the results. In carrying out the test, the bituminous constituents of the asphalt mixture must first be isolated. This is effected in the following manner: 10 grms. of the sample are treated with 75 c.c. of ether, saturated cold with strong hydrochloric acid, added in three to four portions with constant stirring. After about ten minutes, 75 c.c. of water are added, and the mixture is heated to drive off the ether. The residue is filtered off, washed and dried, and then exhaustively extracted with boiling chloroform. The bitumen is recovered on driving off the chloroform, dried for a short time at 105° C., and weighed. Three grms. of this bitumen are treated in a thick test-tube with 6 c.c. of concentrated sulphuric acid, and heated in the boiling water-bath for forty-five minutes with frequent stirring. The product of the reaction is cooled and transferred to a conical flask with about 200 c.c. of water. After about an hour the precipitate settles to the bottom, and the liquid is decanted off through a tared hardened filter-paper on a porcelain funnel, at first without suction. The residue is broken up and washed by decantation until it is brought on to the funnel free from acid; some difficulty may be experienced in filtration, so that, where practicable, a Gooch crucible with a layer of asbestos or treatment in the centrifuge may give better results. The product is dried at 105° C., weighed, and the result calculated as per cent. of the original sample. This result gives, of course, only the percentage of natural bitumen in the sample, and the percentage of natural asphaltum can only be inferred indirectly from the general average of bitumen in the type of asphaltum present. Mixtures prepared from known materials and analysed by the author showed results considerably too low. It is therefore proposed to add 4 as a correction to the percentages found calculated on the original sample, in which case it is shown that numbers approximating very fairly to the correct values are obtained. It is to be noted that the method is only applicable to mixtures of asphaltum with coal-tar pitch, and that mineral oil residues behave on sulphonation like natural asphaltum, so that verification by other tests is necessary to confirm the absence of mineral oil products. J. F. B.

Detection of Natural Asphaltum and Petroleum Pitch in Residues from the Distillation of Coal-Tar. F. Schwarz. (*Chem. Rev. Fett. Ind.*, 1913, **20**, 28-30.)—The distillates from coal-tar consist largely of aromatic hydrocarbons which combine with sulphuric acid to form soluble sulphonic acids, whereas the corresponding distillates from natural asphaltum or petroleum pitch yield only a small proportion of soluble derivatives with sulphuric acid. In the case of mixtures, this test is rendered unreliable by the fact that when a large proportion of asphaltum or petroleum-pitch compounds is present, products capable of combining with sulphuric acid are formed during the distillation. The following modification of the test effects the separation of the compounds that combine with the sulphuric acid and obviates the drawbacks of distillation: 10 grms. of the sample are heated to 160° to 180° C., and mixed for five minutes with 4 c.c. of strong sulphuric acid, and the mixture kept at 180° C. (by means of an oil-bath) until the excess of sulphuric acid has been removed. The residue is then pulverised with 40 grms. of bone charcoal, and the mixture extracted in a Soxhlet apparatus with petroleum spirit of low boiling-point. The extract is evaporated, and the residue taken up with more petroleum spirit, the solution filtered and evaporated, and the residue weighed. Sometimes sulphur derived from the sulphuric acid is present, and in such cases the residue should be washed (without agitation) with acetone, the solution filtered and evaporated, and the residue weighed. The results thus obtained with eleven samples of coal-tar pitch, seven of natural asphaltum, and twelve of petroleum pitch, were as follows:

	Substances not attacked by Sulphuric Acid. Per Cent.
Coal-tar pitch	0·10 to 0·21
Natural asphaltum	1·0 to 16·0
	(usually 3·0 to 8·0)
Petroleum pitch	6·0 to 36·0
	(usually 15·0 to 30·0)

A result greatly exceeding 0·2 per cent. indicates the presence of one of the other products in coal-tar pitch. The addition of 10 per cent. of petroleum pitch to a sample of coal-tar pitch raised the value from 0·21 per cent. to 0·46 per cent. A sample of English "coal-tar pitch" yielded 3·4 per cent. of substances not attacked by sulphuric acid, but this high result was attributed to the original coal having been mixed with shale before the distillation. C. A. M.

Method for the Estimation of Galactan. K. Miyake. (*J. Coll. Agric. Tokio Univ.*, 1912, **4**, 337-345; through *Chem. Zentralbl.*, 1913, I., 465-466.) —The substance is extracted with ether and then heated on a water-bath with 60 c.c. of nitric acid of sp. gr. 1·15, until the volume of the liquid is reduced to 20 c.c. The mixture is then filtered, and the residue is washed with hot water. The filtrate is evaporated to one-third its volume, and placed aside for twenty-four hours; 10 c.c. of water are then added, and the mixture allowed to stand for a further period of twenty-four hours. The mucic acid which crystallises out is collected on a filter, washed successively with cold water, alcohol, and ether, dried at 100° C., and weighed. The quantity of substance taken for the estimation depends on its galactan-content; when the amount of galactan present is less than 16 per cent., 3 grms. of substance may be taken, and correspondingly less as the galactan-content increases. Should only small quantities of galactan be present, the filtrate must be evaporated to one-fifth its volume and then set aside for several days. The yield of mucic acid is not quite proportional to the amount of galactan, but the average factor 1·33 may be used to convert the weight of mucic acid found into galactan.

W. P. S.

Estimation of Hydrocarbons of the Acetylene and Olefine Series in Mixtures of Gaseous Hydrocarbons. P. Lebeau and A. Damiens. (*Comptes rend.*, 1913, **156**, 557.)—The usual absorbents, ammoniacal cuprous chloride and bromine, are inconvenient in use, and the substitution of potassium mercuric iodide for the absorption of acetylene hydrocarbons, and of sulphuric acid containing a catalyser (vanadic acid, uranic acid, tungstic acid, molybdic acid) for the absorption of olefines, is suggested. The potassium mercuric iodide solution, containing 25 grms. mercuric iodide and 30 grms. potassium iodide in 100 c.c. water, is rendered alkaline before use by the introduction of a fragment of potassium hydroxide into the absorption-tube, and is capable of absorbing twenty times its volume of acetylene. It dissolves ethylene to the same extent as does water, and the gas may be recovered by evacuation. The sulphovanadic acid reagent contains 1 grm. vanadic oxide in 100 grms., 97 to 98 per cent. sulphuric acid, and the sulpho-uranic acid reagent 6 grms. of uranyl sulphate in the same quantity of acid. A mixture of 3·50 c.c. methane, 10·14 c.c. ethylene, 8·88 c.c. acetylene, and 4·88 c.c. allylene, gave 13·53 of residual gas after treatment with the potassium mercuric iodide reagent, instead of the theoretical 13·64; the difference is due to dissolved ethylene. After treatment with the sulphovanadic acid reagent 3·48 c.c. (instead of 3·50 c.c.) of methane were left. O. E. M.

Hexabromide Number of Fatty Oils and the Estimation of Rape Oil in Raw Linseed Oils. A. Eibner and H. Muggenthaler. (*Farbenzeitung*, 1912, **18**, 131, 175, 235, 356, 411, 466, 523, 582, 641; through *Chem. Zentralbl.*, 1913, I., 567.)—The utility of the hexabromide number, the literature of which is discussed exhaustively, for the valuation of linseed oil, depends upon the determination of the linolenic acid, the constituent of most importance in the drying process. This acid gives a hexabromide insoluble in cold ether, while the simultaneously formed tetrabromide of the isolinolenic acid is dissolved. The following method is based on lengthy series of experiments: (1) *Preparation of Pure Linseed Oil Fatty Acids.*—Three portions of linseed oil, each of 3·5 grms., are saponified separately in 220 c.c. evaporating basins on the water-bath, with stirring, by means of 45 c.c. of $\frac{N}{2}$ alcoholic potassium hydroxide solution; the soap is evaporated to dryness, and each portion dissolved in 50 c.c. of warm water, the wash water from one basin being used to dissolve the soap in the next. The united solutions (180 c.c.) are transferred warm to a 1-litre separating funnel bearing marks at 180 and 340 c.c., and, when cool, treated with 20 c.c. 5 N sulphuric acid and 140 c.c. of ether. After agitation and separation, the ethereal solution is allowed to stand all night over 70 grms. dehydrated potassium sulphate, and filtered; the potassium sulphate is washed out several times with dry ether. The ether is distilled off, the last portions being removed by a current of hydrogen purified by bubbling it through an alkaline solution of a lead salt and through concentrated sulphuric acid; the residue is dried in a vacuum over sulphuric acid. The fatty acids are made up to an approximately 10 per cent. solution with dried ether. (2) *Bromination.*—In a wide-mouthed conical flask surrounded by a freezing mixture, 20 c.c. of the ethereal solution, containing 1·9 to 2·0 grms. fatty acids, are cooled to $-10°$ C.; 1 c.c. of bromine is added, 0·5 c.c. in single drops and the rest two drops at a time, with vigorous shaking at each addition, the whole requiring about thirty minutes. A cork is inserted, and the flask allowed to stand for two hours at $-10°$ C. The brown mother liquor is then poured through a weighed asbestos-packed Daniel tube, the precipitate is shaken up with 5 c.c. of ether at $-10°$ C. and allowed to settle in the freezing mixture, and the liquid poured off. The precipitate is then shaken up with 5 c.c. of cooled ether and transferred to the filter, and, after the flask and precipitate have been washed three times with 5 c.c. of ether at $-10°$ C., drained by suction and dried at 80° to 85° C. The hexabromide thus obtained should be white and granular; its melting-point is 177° C.

Average values of the hexabromide number of various raw linseed oils of authenticated origin were found to be: Dutch, 51·73; River Plate, 51·66; East Indian, 50·50; Baltic, 57·96. Baltic oil is thus readily distinguished from the others. The hexabromide number is not materially affected by bleaching, which thus does not limit the applicability of an oil; it is much lower in linseed oil varnishes, and may be reduced to zero in oxidised oils. Other oils gave: poppy and china-wood, 0·0; perilla, 64·12; ocumi, 60·98; rape, 6·34; soya bean, 7·17.

Estimation of Rape Oil in Linseed Oil.—The erucic acid test having revealed the presence of rape oil, the hexabromide number shows the amount of adulteration. The addition of 10 per cent. of rape oil lowers it by approximately 4·4, and consequently, the origin of the oil being taken into account, the amount of the addition can be calculated. An allowance of 4 per cent. for the permitted contamination of the flax seed with seeds of cruciferous plants is then deducted. O. E. M.

Oxygen Absorption Test for Linseed Oil. H. Mannhardt. (*J. Ind. and Eng. Chem.*, 1913, **5**, 129-131.)—The recent report of a subcommittee of the American Society of Testing Materials is said to be quite misleading, because the four members of the committee disregarded the direction of earlier experimenters to use a film of approximately uniform thickness in all experiments. Glass plates of uniform size were used, but one experimenter used anything from 0·07 to 0·40 grm. oil, another anything from 0·17 grm. to 2·73 grms. It is not surprising, therefore, that the results of the committee's work seem to discredit the oxidation test as untrustworthy. The author has used several forms of the test and found them trustworthy. One method recommended is that in which about a gram of oil is weighed out and mixed with about 10 grms. of finely ground natural silica, contained in a shallow vessel of about 6 cm. diameter. The use of 5 c.c. of ether greatly facilitates the mixing. Aluminium plates also serve well for the test, two drops of oil being applied to each side of each of five plates (3 inches by 6 inches) and distributed over it by means of the finger, the whole series being suspended in a frame of aluminium wire, so as to allow of their being weighed on the fine balance. The time required to reach maximum weight is at least as important as the increase of weight itself. G. C. J.

Solidified (Hydrogenised) Oils and the Detection of Arachidic Acid. H. Kreis and E. Roth. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1913, **25**, 81-85.)—Sesame, earthnut, and cottonseed oil which have been solidified by treatment with hydrogen in the presence of a catalyst (*cf.* ANALYST, 1912, **37**, 452) yield a reaction with Bellier's test (nitric acid and resorcinol in benzene solution), whilst solidified whale oil gives an orange coloration with the test. The sesamol present in sesame oil is not affected by hydrogenisation, and solidified sesame oil gives the reactions described by Kreis for ordinary sesame oil (ANALYST, 1903, **28**, 364). Arachidic acid may be detected in mixtures of solidified earthnut oil with ordinary or other solidified oils by saponifying 20 grms. of the oil under examination with 40 c.c. of alcoholic potassium hydroxide solution (20 per cent.), adding 60 c.c. of alcohol, and acidifying the mixture with 50 per cent. acetic acid; the mixture is then treated at a boiling temperature with 1·5 grms. of lead acetate dissolved in alcohol, and after the lapse of about twelve hours the precipitated lead soap is separated, and decomposed by heating with hydrochloric acid. The fatty acids thus obtained are crystallised three times from alcohol (90 per cent.), and the melting-point of the resulting crystals is determined. Should the oil contain not less than 5 per cent. of earthnut oil the melting-point will lie above 70° C. W. P. S.

Lemon-Grass Oils from India. (*Bull. Imp. Inst.*, 1912, **10**, 546-548.)—Four samples of lemon-grass oil from Assam gave the following analytical results:

	1.	2.	3.	4.
Specific gravity at 15°/15° C. ...	0·9039	0·9035	0·8973	0·9109
Optical rotation in 100 mm. tube at 20° C.	- 0°30′	- 0°40′	- 0°20′	—
Citral (by sodium bisulphite method) per cent.	72·2	81·5	78·0	73·7

None of the oils dissolved to a clear solution, even in 10 or more volumes of 70 per cent. alcohol. In the case of No. 2, which was described as "Cochin" oil, the incomplete solubility was attributed to the presence of other grasses in the lemon grass distilled. C. A. M.

Tests on the Opacity and Hiding Power of Pigments. G. W. Thompson. (*J. Ind. and Eng. Chem.*, 1913, **5**, 120-123.)—Hiding power is defined as the power of a paint to obscure optically a surface painted with it, opacity as the obstruction to the direct transmission of visible light afforded by any substance, comparison being made with sections of equal thickness. Opacity refers to tests made under standard conditions, whilst hiding power refers to tests made of paints as they are used. The distinction becomes clearer when considered with reference to a paint, the opacity of which is measured with a standard thickness of paint; whilst in the case of hiding power, the thickness of the paint will vary according to the spreading rate at which the paint is applied. Arguments are brought forward in support of the view that, in all comparisons of opacity, the relative *volume* of the pigment (not its weight) should be considered, and that a standard of opacity should be based on a definite volume of the pigment placed in a definite volume of the menstruum. Except for zinc oxide and other pigments which need further dilution for testing by the author's method, he uses a mixture of 1 part by true volume of pigment to three parts of oil. The accuracy of earlier photometric methods (*e.g.*, the photographic method of Hürter and Driffield, *J. Soc. Chem. Ind.*, 1890, **9**, 455) depends upon no light being reflected by the object being tested, or on the reflecting power of the object being accurately known. Considering opacity as having to do only with the light which is being transmitted and not at all with the light reflected from the surface of the paint, the author has devised an apparatus, not shown in detail in the paper, by which the opacity of a paint may be determined as follows: Paint is placed between two glass surfaces, the distances between which can be controlled and measured by a micrometer screw, and the film reduced in thickness by bringing the plates closer together, until the light passing through exactly matches that passing through a standard plate or paper, the opacity of which has been tested on a photometer bench. A less opaque standard is then substituted for the first, and the plates are again brought closer together until a match is obtained. From Bouguer's logarithmic law that if a given layer absorbs a certain fraction of transmitted radiation, the next equal layer will absorb the same fraction, it follows that the opacity, as defined, of the paint can be deduced from the thickness of the two films and the known opacity of the standards. The coefficient of opacity is defined as the proportion of light, expressed as a decimal fraction of unity, absorbed during transmission through a thickness of 0·01 mm. of paint. G. C. J.

Testing Parchment Papers. V. Fortini and A. Ceccherelli. (*Chem. Zeit.*, 1913, **37**, 237-239.)—The following tests are said to be more conclusive and more easily applied than chemical or microscopical tests for distinguishing between genuine and imitation parchment papers. The first test described consists in determining the tensile strength of the paper before and after half an hour's immersion in water at 75° to 80° C. The tensile strength is always reduced by the treatment, but with genuine parchment papers the ratio of the initial to the final value is always less than 2 : 1, whereas with imitation parchment papers it is always greater than 10 : 1. The second test described also depends on the relative indifference of genuine parchment papers to wetting by hot water. It is proposed because apparatus for determining tensile strengths is found in comparatively few laboratories, whereas Vicat's needle is more commonly met with. The paper, before and after treatment with hot water, is stretched in a 10-cm. frame, covered with a disc of metal with a central hole through which the Vicat needle (of 1 cm. diameter) can just pass freely, and the needle is then loaded with shot until the paper breaks. The breaking loads do not measure any property of the paper in absolute units, as the critical load is dependent, among other things, on the diameter of the frame on which the paper is stretched; but, as in the measurements of tensile strength, the ratio of the load required to break genuine parchment paper before and after treatment with hot water is always less than 2:1, whereas with imitation parchment papers it always exceeds 10:1. G. C. J.

Estimation of Phenol in Crude Carbolic Acid and Tar Oils. J. M. Weiss. (*J. Franklin Inst.*, 1912, **174**, 683; through *Chem. Zentralbl.*, 1913, I., 565.)—The m.-pt. and sp. gr. suffice for the determination of phenol in crude carbolic acid and tar oil; the b.-pt. is unnecessary. Tables showing the phenol content of mixtures of phenol and cresols for m.-pts. from 0° to 23° C. and upwards, and for various densities, are worked out. O. E. M.

Crude Petroleum from the Gold Coast. (*Bull. Imp. Inst.*, 1912, **10**, 579-583.)—Samples of crude petroleum from Bonyere, in the Gold Coast Colony, were black, viscid liquids, which on distillation left from about 14 to 20 per cent. of bituminous pitch. The distillates consisted mainly of lubricating oils, with a small proportion of kerosene. C. A. M.

Estimation of Soaps in Lubricating Greases. J. Marcusson. (*Chem. Rev. Fett. Ind.*, 1913, **20**, 43-45.)—The volumetric method of estimating the amount of soap in lubricating greases from the results of a titration of the free and combined fatty acids is very difficult in the case of dark-coloured greases, and requires special precautions when rancid fats are present. A method more generally applicable has been based upon the facts that acetone dissolves only a very small amount of the soaps of fatty acids, and that the soaps may then be separated from insoluble inorganic substances (graphite, etc.) by treatment with a mixture of benzene and alcohol. Ten grms. of the sample are digested for several hours with 100 c.c. of cold acetone (freshly distilled), the mass being repeatedly broken up with a glass rod until in a very fine state of division. The solution is filtered, and the residue washed with acetone and then boiled with a mixture of benzene and alcohol (9 : 1). The hot solution of soap is filtered and evaporated, and the residue dried at 105° C. until constant in weight. The results thus obtained are occasionally higher than those given by the older volumetric method, probably owing to the presence of considerable quantities of water-soluble fatty acids which escape estimation. Certain very viscous oils do not dissolve readily in acetone, and in such cases the last traces of fatty oil may be removed from the insoluble residue by washing it with a mixture of acetone and petroleum spirit boiling at 50° C. (3 : 1). Alkali soaps may be separated from calcium or aluminium soaps by boiling the mixed soaps with 50 per cent. alcohol.

For a quantitative estimation this is best done prior to the separation of the inorganic constituents. The pitch-like distillation residues introduced into certain inferior greases are partially soluble in acetone, and soluble in benzene-alcohol, so that the method is not applicable in such cases. These greases, however, are not often met with. As a rule, only an insignificant amount of soap is dissolved by the acetone, and no correction is usually required. Calcium resinate, however, is fairly soluble, and when it is present the amount dissolved by the acetone may be found by estimating the ash and calculating the corresponding amount of resin acids of mean molecular weight 300. The substances soluble in the acetone may include petroleum oil, resin oil, tar oil, fatty oils, and glycerol, the last of which generally separates as a viscous layer below the oils. C. A. M.

Contribution to Methods of Determining Nitrogen in Humus. C. B. Lipman and H. F. Pressey. (*J. Ind. and Eng. Chem.*, 1913, **5**, 143-144.)—Importance attaches to the nitrogen determination in humus, since it is this, and not the total soil nitrogen, which can be depended upon to yield through nitrification an available supply of nitrogen to plants. The author finds that the method of Hibbard (*J. Ind. and Eng. Chem.*, 1910, **2**, 463) is more satisfactory than any of the other modifications of the Kjeldahl method, in all cases but one yielding the highest nitrogen figure. Other advantages claimed are freedom from bumping, and rapidity and ease of manipulation. The material is first freed from ammoniacal nitrogen by boiling with magnesium oxide, and is then digested with 30 c.c. of strong sulphuric acid until fumes of sulphur trioxide are evolved. Twelve grms. of a mixture consisting of 10 grms. potassium sulphate, 1 grm. ferrous sulphate, and 0·5 grm. copper sulphate, are then added, and digestion continued until completed. The other methods tested included Wilfarth's, Gunning-Attenberg, and the salicylic acid, this last proving next best to Hibbard's. H. F. E. H.

Comparison of Some Qualitative and Quantitative Methods for Carbonates in Soils. E. W. Gaither. (*J. Ind. and Eng. Chem.*, 1913, **5**, 138-143.)—The method devised by Marr (Analyst, 1910, **35**, 77) was investigated, and his results confirmed by the author, who used the double titration method of Brown and Escombe (*J. Agric. Sci.*, 1905, **1**, 322). Over a hundred soils were tested, and the results compared with their litmus-paper reactions and with the Veitch qualitative test, in which 10 grms. of the soil are extracted in the cold with 100 c.c. of water, allowed to settle, and 50 c.c. pipetted off; phenolphthaleïn is then added, and the solution is boiled down to about 15 c.c., the degree of colour (if any) being recorded as slightly alkaline, etc. For the litmus-paper test, strips of red and blue azolitmin paper were either placed direct on the moistened soil or with filter-paper intervening, and left covered up over night.

It is shown that the official method of boiling the soil with mineral acids at 100° C. decomposes organic matter and liberates carbonic acid, which was not present as a carbonate, but that the method used by Marr (*loc. cit.*) of boiling the soil under reduced pressure at 50° C. with very dilute mineral acid is quite satisfactory. The litmus-paper test is the best qualitative test for native carbonates in soils from humid regions. Some soils will, however, give alkaline aqueous solutions not due to the presence of carbonates, but to the hydrolysis of certain minerals in such soils. Although the reddening of blue litmus-paper by soils may be due to the absorption of the base from the hydrolysed litmus salt, the presence of native carbonates may either prevent this selective absorption or cause an interchange of bases to take place. If a native carbonate, capable of being decomposed by weak mineral acid at low temperatures, is present in a soil, it is indicated by the bluing of red litmus-paper; but if there are no alkalies or basic materials capable of giving alkaline solutions, their absence will be indicated by the reddening of blue litmus-paper.

H. F. E. H.

Analysis of Spirit Varnishes. Part I.: Investigation of the Solvent. H. Wolff. (*Farbenzeitung*, 1912, **18**, 856, 910, 950; through *Chem. Zentralbl.*, 1913, I., 853.)—The sample is heated on the water-bath, under a reflux condenser, with ten times its volume of water, until the resin forms a coherent mass. When cold, the liquid is poured off and the cake of resin boiled up twice with a small quantity of water. The combined aqueous liquid is distilled until a volume equal to that of the varnish taken is obtained, and the distillate is shaken vigorously with salt solution; if homogeneous, the liquid contains only ethyl alcohol, methyl alcohol and alcohol; ether, benzine, benzene, amyl alcohol, ethyl and amyl acetate, and turpentine separate in an upper layer. From the distillation flask a volume equal to that of half the sample is distilled, added to the salt-containing portion (see above), the combined solutions distilled up to 100° C., and the distillate made equal in volume to the varnish taken. Acetone is tested for by the sodium nitroprusside reaction, and estimated by adding 10 c.c. of 10 per cent. sodium hydroxide solution and 50 c.c. $\frac{N}{10}$ iodine to 1 c.c. of the distillate, acidulating after three minutes with dilute sulphuric acid, and titrating back the iodine (1 c.c. iodine solution is equivalent to 0·967 mgrm. acetone). For the estimation of the methyl alcohol, 1 to 5 c.c. of the alcohol are dropped into a cooled mixture of 20 c.c. 10 per cent. potassium dichromate solution and 10 c.c. concentrated sulphuric acid, allowed to stand in a closed flask for one to two hours, a volume equal to that of the alcohol taken distilled off, and the distillate heated in a sealed tube with 1 grm. dimethyl aniline and 1 c.c. of 1 in 5 sulphuric acid for thirty minutes in the water-bath. When cool, the liquid is rendered alkaline with sodium hydroxide, heated with water until the smell of dimethylaniline is removed, shaken with lead peroxide, acidulated with acetic acid, filtered, allowed to stand for five minutes, and the tint estimated colorimetrically. Comparative experiments with mixtures of alcohol and methyl alcohol should be carried out. The layer above the salt solution is treated as follows: 1 c.c. is allowed to stand twenty-four hours with 25 c.c. $\frac{N}{1}$ alcoholic potassium hydroxide, and is titrated back. If more than 0·2 to 0·4 c.c. of potassium hydroxide is neutralised, 1 c.c. represents about 5 vols. per cent. acetic ester, or 7·5 per cent. amyl acetate. The presence of amyl acetate or amyl alcohol in any quantity is revealed by the smell. An aliquot portion of the liquid is slowly poured, with cooling, into double the volume of sulphuric acid (sp. gr. 1·80). If no separation occurs, benzine, benzene, and any considerable quantity of turpentine are absent (turpentine colours the acid dark brown);

if the liquid separates completely, it contains benzine, or benzene or its homologues. To test for esters, an aliquot part of the liquid is saponified by allowing it to stand with concentrated alcoholic potassium hydroxide, diluted to five times its volume with water, and half distilled off. The distillate is salted out with sodium chloride, and the upper layer poured into sulphuric acid (sp. gr. 1·80). If there is no separation on salting out the distillate, the original liquid was exclusively acetic ester. Separation of a layer mostly soluble in water shows the presence of ether or amyl alcohol, while solubility in sulphuric acid reveals amyl alcohol, ether, or turpentine, benzine, and benzene. If nothing goes into solution, benzine or benzene is indicated; if everything, amyl acetate, originally present as such or as amyl acetate. Examples of the method, and hints for the detection of solvents containing chlorine, are given. O. E. M.

Turpentine Oils from India. (*Bull. Imp. Inst.*, 1912, **10**, 539-546.)—The bulk of the turpentine oil produced in India is derived from *Pinus longifolia*, but oil could also be obtained from other species of Indian pines, including *P. Khasya* and *P. excelsa*. The oil from the last, after redistillation to remove the yellow colour, is equal in quality to the best French and American oils. The following results were obtained with samples of these different oils:

Turpentine Oil from—	Sp. Gr. at 15°/15° C.	Optical Rotation in 100-mm. Tube.	FRACTIONAL DISTILLATION.								
			165° to 170° C.		170° to 175° C.		175° to 180° C.		180° to 190° C.	190° to 195° C.	Residue and Loss. Per Cent.
P. longifolia:			Per Cent.	Rotation.	Per Cent.	Rotation.	Per Cent.	Rotation.	Per Cent.		
Crude ...	0·868	+0°20′	43	−5°25′	40	+2°0′	8·0	+8°5′	3·0	—	6
Refined ...	0·866	−0°40′	56	−4°40′	33	+3°0′	4·5	—	1·5	—	5
			157° to 158° C.		158° to 160° C.		160° to 170° C.				
P. excelsa ...	0·862	+36°40′	74	+37°10′	16	+36°15′	7	+34°0′	—	—	3
			162° to 163° C.		163° to 165° C.		165° to 169° C.				
P. Khasya...	0·870	−4°50′	25	−1°50′	57	−4°40′	11	−10°40′	-	—	7 (rotation −7°30′)

C. A. M.

Estimation of Water in Petroleum and its Products. J. C. Allen and W. A. Jacobs. (*Techn. Paper 25. Dept. of the Interior, Bureau of Mines, U.S.A.*, 1912, 1-11.)—An outline is given of the different methods of estimating water in petroleum products, together with a critical discussion of the accuracy of each method. Estimation of the water by ascertaining the loss on heating gives approximate results, and is only applicable to heavy oils and greases. The method of diluting the oil with a solvent and allowing the water to separate by gravity is also approximate, and can only be used with thin oils; whilst the use of centrifugal force in this method increases the rapidity of the separation and gives somewhat more accurate results. The calcium carbide method, in which the amount of water is calculated from the volume of acetylene evolved, is convenient to use, and in the case of petroleum products gives results that are accurate within about 3 per cent. of the real values. Graefe's method (*Petroleum*, 1906, **1**, 818) of shaking the oil with finely-cut sodium, and measuring the volume of hydrogen evolved, gives more accurate results than the carbide method, since hydrogen is not appreciably soluble in petroleum products. Comparison of the sample with standard mixtures of oil and water gives only roughly approximate results; and the same criticism applies to the method in which the sample is shaken with standard acid, and the decrease in acidity of the aqueous layer estimated. The electrical process of Cottrell and Wright (*U.S. Pat.* 987114 of 1911), in which a current is passed through the emulsified oil, and the separated water drained off through a cheesecloth into the lower end of the tube, will rapidly reduce the amount of water down to 0·5 to 1 per cent. with little heating. The residual quantity can then be eliminated by one of the other methods. Distillation of the oil with an immiscible solvent such as benzene or toluene, and measurement of the volume of water separating from the distillate (*cf.* ANALYST, 1907, **32**, 21), is accurate to within about 0·033 grm. of water per 100 c.c. of benzene and oil in the distillate. The method of direct distillation of the water is also fairly accurate. The distillation of 200 c.c. of the sample should be carried out at temperatures between 90° and 150° C. at the rate of about 1 drop per second, and the water removed from the receivers by means of a micro-pipette and weighed, the residual traces in the condenser tube being absorbed by means of a tared pellet of cotton wool. When much water is present, a slow current of carbon dioxide passed through the oil will prevent bumping and overheating. The method gives results accurate to within about 0·003 grm. of water in the distillate, provided that the water is cooled to about 2° C. C. A. M.

INORGANIC ANALYSIS.

Iodic Acid Method of Estimating Bromine in Halogen Salts. F. A. Gooch and P. L. Blumenthal. (*Zeitsch. anorg. Chem.*, 1913, **80**, 36-42.)—Bugarski's method for the separate estimation of bromide and chloride when occurring together (ANALYST, 1896, **21**, 79) always underestimates bromide and overestimates chloride. The error, which varies from almost nil to 3 per cent., according to the exact experimental conditions, is shown to be due in all probability to the formation of iodine chloride and bromide. G. C. J.

Estimation of the Carbonates and Hydroxides of Potassium and Sodium when together in Solution. W. A. Bradbury and F. Owen. (*Chem. News*, 1913, **107**, 85-86.)—In order to determine the composition of a "boiler fluid" containing the hydroxides and carbonates of sodium and potassium, an attempt was made to separate the hydroxides from the carbonates by extraction with absolute alcohol, but this was found impracticable owing either to carbonation of the hydroxides during evaporation of the original solution; or, if this were obviated by concentrating out of contact with air, the residue in the flask after alcoholic extraction was found to contain much hydroxide along with the carbonate. It is unnecessary to effect a separation such as that attempted, since it is possible to obtain the desired results by calculating from three values: (1) The acid equivalent to the carbonates present; (2) the acid equivalent to the hydroxides; and (3) the total weight of mixed sulphates left after evaporating to dryness a portion of the solution rendered just neutral to methyl-orange by titration with standard sulphuric acid. The values (1) and (2) are obtained in the usual way by titration first with phenolphthaleïn, and then with methyl-orange. Having obtained these three values, the total acid required is calculated to its equivalent of potassium sulphate, and from this is subtracted the weight of the mixed sulphates; the difference is due to the sodium sulphate. The weights of potassium and sodium sulphate present in a given volume of the neutralised solution can therefore be calculated. As the whole of the acid is calculated to potassium sulphate, and since the acid was, in fact, neutralised by carbonates and hydroxides, it is evident that the proportion of the total sulphate due to carbonates and hydroxide is equivalent to the amount of acid used for each respectively; therefore the proportion of the above obtained difference due to the carbonates and hydroxides respectively is also proportional to the amount of acid used for each. A worked example of a particular case is appended, which makes the above reasoning clear.

H. F. E. H.

Detection of Traces of Copper. W. B. Pritz, A. Guillanden, and J. R. Withrow. (*J. Amer. Chem. Soc.*, 1913, **35**, 168-173.)—For determining whether all the copper has been deposited from a solution submitted to electrolysis, the following test is recommended: One c.c. of the electrolyte is transferred by means of a pipette to a colourless, thin glass tube, about 3 to 5 mm. in diameter and 15 cm. long, and with the side walls coated with black paper. The solution is made alkaline with ammonia, then acid with acetic acid, and two drops of 2 per cent. potassium ferrocyanide are then added. The test will just detect 0·1 mgrm. of copper in 100 c.c. of electrolyte. Other modifications of the ferrocyanide test differ greatly in sensitiveness, and, in particular, attention is called to the fact that the sensitiveness is approximately inversely proportional to the ferrocyanide concentration. With a nitric acid electrolyte, the pink colour of copper ferrocyanide rapidly gives place to, or is entirely masked by, a yellowish-green colour, but this interfering colour does not appear in solutions containing a negligible amount of copper ($<$ 0·1 mgrm. per 100 c.c.). In presence of zinc the method fails, and ammonium sulphide must be used, the limiting sensitiveness with freshly prepared solutions being 0·15 mgrm. per 100 c.c. Solutions of ammonium sulphide deteriorate so rapidly on storage that for this purpose they should be freshly prepared each day. A solution a fortnight old will not detect 3 mgrms. of copper in 100 c.c.

G. C. J.

New Hydrogen Table. L. Vanino and A. Schinner. (*Zeitsch. angew. Chem.*, 1913, **26**, 55-56.)—A table is given showing the weight of 1 c.c. of hydrogen in fractions of a mgrm., at temperatures ranging from 10° to 25° C., and barometrical pressures of 700 to 770 mm.

C. A. M.

Application of Diphenylcarbazide ("Diphenylcarbohydrazide") as Indicator in Titration of Iron with Dichromate. O. L. Barnebey and S. R. Wilson. (*J. Amer. Chem. Soc.*, 1913, **35**, 156-162.)—An investigation of Brandt's proposal

(*Zeitsch. anal. Chem.*, 1906, **45**, 95) to use diphenylcarbazide as internal indicator in dichromate titrations. The end-point is excellent, but the indicator itself is a reducing agent and a blank titration with some ferric chloride and a measured quantity of indicator, equal to that used in ordinary titrations, is necessary. Iron ores (0·5 grm.) are dissolved in 20-25 c.c. hydrochloric acid with the addition of a small excess of stannous chloride, which is destroyed by means of mercuric chloride, and the ferrous solution is diluted to 500 to 600 c.c. About 20 c.c. of a manganese solution, containing 100 grms. manganese sulphate, 140 c.c. concentrated sulphuric acid, and 50 c.c. of phosphoric acid (sp. gr. 1·7) per litre, is next added, and followed by 10 c.c. of hydrochloric acid, and a measured volume (2 to 3 c.c.) of the indicator, which is made by dissolving 1 grm. of diphenylcarbazide in acetic acid and diluting to 1,000 c.c. Directions are given for preparing diphenylcarbazide. The first drop of dichromate produces a pink or red tinge, which becomes deep red as the titration proceeds. The colour gradually changes to lavender as more and more chromic chloride is produced, and finally gives place to the clear green colour of that salt, the end-point, as stated, being quite sharp. G. C. J.

Application of Electric Resistance Furnace to Determination of Oxygen in Iron and Steel. R. H. McMillen. (*J. Ind. Eng. Chem.*, 1913, **5**, 123-125.)—With the reservation that the oxides of tungsten and aluminium and some other oxidised constituents of steel are not reduced by Ledebur's method (see also Walker and Patrick, ANALYST, 1912, **37**, 586), the following modification of that method is recommended. Its most original feature is the use of a second combustion tube to remove traces of oxygen from the hydrogen employed. The hydrogen generated from pure hydrochloric acid and zinc is passed through scrubbers containing a 25 per cent. alkaline solution of pyrogallol, 50 per cent. caustic potash, stick potash, soda lime, and pumice saturated with concentrated sulphuric acid. It then passes through the first combustion tube, which contains several spirals of platinum gauze, and is maintained at 950° C., and thence through a U-tube containing phosphoric anhydride and glass beads to the second combustion tube, maintained at the same temperature as the first, and containing in a porcelain boat 25 grms. of drillings (through 20 and over 30 meshes to the lineal inch). The water is collected in a U-tube filled with phosphoric anhydride kept open by admixture with glass beads, and a Drexel wash-bottle containing concentrated sulphuric acid serves as guard-tube and completes the chain of apparatus. The combustion tubes used are thick-walled quartz tubes of $\frac{7}{8}$ inch internal diameter and 24 inches long, the furnace being of the type now common in steel works for determination of carbon (*e.g.*, see Blount and Levy, ANALYST, 1909, **34**, 94). G. C. J.

Determination of Lithium. W. W. Skinner and W. D. Collins. (*U.S. Dept. Agric., Bureau of Chem., Bull. No.* 153, September 14, 1912; through *J. Soc. Chem. Ind.*, 1913, **32**, 214.)—As a result of an investigation of various methods which have been proposed for the determination of lithium in mineral waters that recommended by Gooch, modified slightly in some of the details of manipulation, was found to be satisfactory when weighable amounts of the metal are present. The method is carried out as follows: The water is acidified with hydrochloric acid, barium chloride is added, and the whole is evaporated to dryness; the residue is dissolved in a small quantity of water and hydrochloric acid, the barium sulphate is removed by filtration, and the filtrate is evaporated. After ignition to remove ammonium salts, the residue is dissolved in water, the magnesium salts are precipitated by the addition of milk of lime (a double precipitation is necessary if the water contains a large quantity of alkalis), and the excess of calcium hydroxide, together with barium salts, is removed by treatment with ammonium carbonate in ammoniacal solution. The filtrate from the calcium precipitate is evaporated, the residue is ignited, and any remaining traces of calcium salts are removed as oxalate. The solution is then evaporated in a weighed platinum basin, the residue is treated with a small quantity of hydrochloric acid, again evaporated to dryness, ignited, and weighed. The mixed chlorides thus obtained are dissolved in a few c.c. of water, and the solution is transferred to a flask (any insoluble matter is filtered off, ignited, and its weight deducted from that of the mixed chlorides); the solution is now boiled until the salts begin to crystallise out, and about 40 c.c. of amyl alcohol are added. The contents of the flask are next distilled until a thermometer in the neck of the flask indicates 130° to 132° C. (the boiling-point of amyl alcohol), a drop or two of dilute hydrochloric acid being then added, and the distillation continued until the boiling-point of amyl alcohol is again reached. The mixture is cooled slightly and filtered, note being made of the volume of the filtrate, and, after the insoluble portion has been washed with a little anhydrous amyl alcohol, the filtrate is evaporated to dryness. The residue, consisting of lithium chloride with small amounts of potassium and sodium chlorides, is treated with sulphuric acid, ignited, and weighed. The sulphates are then dissolved in water, and treated with ammonium phosphate and ammonia; if any precipitate forms within about fifteen hours, it is collected and weighed as magnesium pyrophosphate, an equivalent weight of magnesium sulphate being subtracted from the weight of the mixed sulphates. To correct for the solubility of potassium and sodium chlorides in amyl alcohol, there must be subtracted, from the weight of the sulphates, 0·00109 grm. for every 10 c.c. of the amyl alcohol filtrate (exclusive of the amount used for washing). In many cases the corrections amount to the total weight obtained within the limit of the weighing error of the work, and the amount of lithium present may then be determined by means of the spectroscope. For this purpose the residue of sulphates is dissolved in water and spectroscopic observations are made, until, with increasing dilution of the solution, the lithium line is no longer visible. Knowing the extent of the dilution, the amount of lithium is ascertained by comparison observations with solutions containing known quantities of lithium under the same conditions as to dilution, etc. The limit of visibility of the red line of the lithium spectrum is reached with 0·00002 mgrm. of lithium per c.c. of solution. A preliminary and approximate spectroscopic determination of lithium in the mixed chlorides obtained in water analyses saves much time by indicating whether it is worth while to extract the lithium for weighing. A similar test may be used to determine whether much strontium is present in the calcium precipitate obtained in the usual way.

Volumetric Determination of Manganese in Rocks, Slags, Ores, and Spiegels. F. J. Metzger and L. E. Marrs. (*J. Ind. and Eng. Chem.*, 1913, **5**, 125-126.)—The method previously described by the authors (ANALYST, 1911, **36**, 368) has proved particularly useful for the determination of small quantities of manganese in silicate rocks. From 2 to 5 grms. of the material are boiled with 5 to 15 c.c. of dilute (1 : 2) sulphuric acid, to which 5 to 15 c.c. of hydrofluoric acid are subsequently added, and boiling continued until complete decomposition is effected. From 5 to 10 c.c. of dilute (1 : 1) nitric acid and 2 to 3 c.c. of concentrated sulphuric acid are next added, the flame removed, and about 1 grm. of ammonium persulphate is added in small portions. When gas ceases to be evolved, the mixture is evaporated to fuming, cooled, diluted with 50 c.c. water, boiled, again cooled, and transferred to a wax beaker. Ammonium fluoride (5 grms.) and 25 c.c. hydrofluoric acid are next added to the mixture, which should measure about 100 c.c., and the manganese titrated with $\frac{N}{50}$ permanganate (Mn titer = 0·787 Fe titer). Slags are treated in a similar manner, but less substance is taken, or the final solution is made up to known volume, and an aliquot portion taken for titration with $\frac{N}{10}$ permanganate. If much manganese is present, the solution obtained on evaporating to fuming with sulphuric acid may contain permanganate. If a pink solution is obtained, the colour should be destroyed by the cautious addition of hydrogen peroxide after the usual process of cooling, dilution, boiling, and re-cooling. The excess of hydrogen peroxide is destroyed by boiling for ten minutes, and the titration completed as usual.

Ores (0·5 to 1·0 grm.) are dissolved in the least possible quantity of hydrochloric acid, and the solution evaporated to fuming after addition of 15 c.c. of dilute (1 : 2) sulphuric acid. The solution is then treated with nitric acid and persulphate, and the process completed in the manner already described. Some siliceous ores and slags may need treatment with hydrofluoric acid as described for rocks.

Spiegels and ferromanganese (0·2 to 1 grm.) are dissolved in 5 to 10 c.c. dilute (1 : 1) nitric acid, and the solution is treated with persulphate after cooling slightly, and then evaporated with sulphuric acid. Nitric acid up to 2 c.c. of concentrated acid in 150 c.c. has no effect on the titration, but very small amounts of hydrochloric acid interfere. It is recommended to have not much more than 60 mgrms. of manganese present in the portion taken for titration, and to use $\frac{N}{10}$ permanganate with spiegels and other relatively high-percentage manganese products. These directions put a limit on the accuracy with which a high-grade ferromanganese can be assayed by the method, but the test numbers given are good. G. C. J.

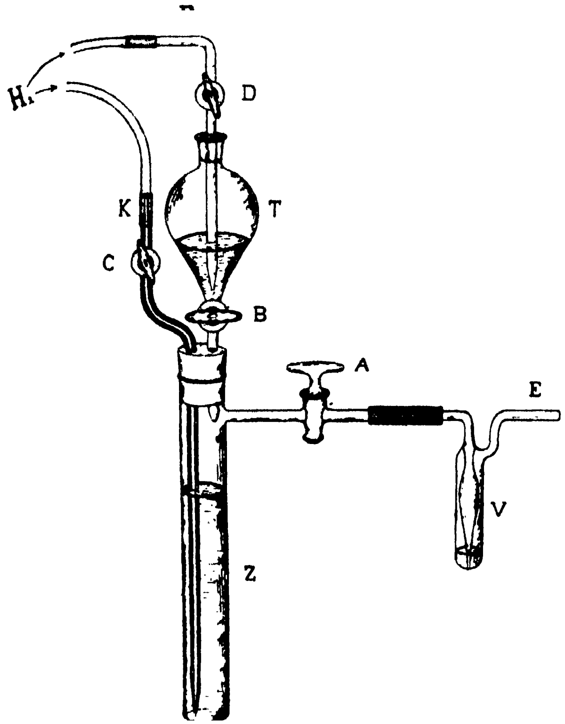
Detection of Nitrate in Presence of Nitrite. W. N. Ivanov. (*Chem. Zeit.*, 1913, **37**, 157.)—By the following method 1 part of nitrate may be detected in presence of 100 parts of nitrite, without risk of the formation of nitric acid by decomposition of nitrous acid. Though less sensitive than the diphenylamine reaction, it has the advantage that only chlorine interferes, ferric salts, even in presence of much nitrite, having no perceptible influence. The reagent is a 0·025 per cent. solution of quadrivalent iridium, made by treating the necessary quantity of IrO_2 or $(NH_4)_2IrCl_6$ with 3 to 5 c.c. of water, then adding 100 c.c. of 98 to 99 per cent. sulphuric acid, and boiling until the solution is colourless. It is important that the concentration of the sulphuric acid in the reagent when bottled for use shall not fall far outside the limits 96 to 96·5 per cent. The test for nitrate is made as follows: Into a stoppered test-tube, with inlet and outlet tubes for the introduction of an indifferent gas, 5 c.c. of the reagent is introduced, a current of carbon dioxide passed through the tube, and the reagent heated to incipient boiling. The flame is then withdrawn, the stopper removed, the solid substance to be tested introduced, and the stopper replaced. In presence of 0·1 mgrm. of nitric acid an evanescent blue appears, whilst 0·5 mgrm. gives rise to a permanent blue, the depth of which is said to be proportional to the amount of nitric acid present. G. C. J.

Estimation of Nitric Oxide. Koehler and Marqueyrol. (*Bull. Soc. Chim.*, 1913, **13**, 69-72.)—The method of Baudisch and Klinger (ANALYST, 1913, 45) is criticised on the ground that it is not available in presence of carbon dioxide. The authors have for many years employed a somewhat similar method, but, in place of the potash used by Baudisch and Klinger, they use a small quantity (0·6 c.c.) of a liquid secondary amine such as monoethylaniline. Monoethylaniline absorbs only its own volume of carbon dioxide at 15° C. and 760 mm. Consequently, if the percentage and partial pressure of carbon dioxide in the gas mixture is small, the amount of carbon dioxide absorbed is negligibly small and, even when much carbon dioxide is present, a correction can be made which reduces the error to negligible proportions. G. C. J.

Notes on Valuation of Ores and Minerals and on Metallurgical Calculations. G. T. Holloway. (*Trans. Inst. Min. and Met.*, 1912, 567-592.)—The paper includes a table of factors for shortening the calculations which must precede any attempt to value an ore. The actual monetary value of the ore depends on a number of considerations beyond the simple chemical relationships set out in the table, for example, presence of undesirable constituents and physical characteristics such as porosity, a desirable property in chemical pyrolusite, or tendency to disintegrate under ordinary conditions of transportation, nearly always a disadvantage. The paper does not attempt to explain how the actual value of every ore, or even every common ore, is to be arrived at. Its object is rather to point out the factors which may affect the value and to encourage the mining man to study these and attempt an independent valuation of his produce, instead of being content with the highest offer he can get. As examples, the valuation of manganese ores, tin ores, and bauxite are discussed in detail, and the discussion which followed the paper led to the publication of further useful notes on the valuation of manganese ores. G. C. J.

Estimation of Phosphoric Acid Soluble in Citric Acid in Thomas Slag. W. Simmermacher. (*Chem. Zeit.*, 1913, **37**, 145-146.)—It is pointed out that, when ferric chloride is added to a phosphoric acid solution containing dissolved silica in order to prevent precipitation of the latter on the addition of ammonium citrate and magnesia mixture, the latter reagents should be added slowly and with constant stirring, otherwise a quantity of ferric hydroxide will be precipitated together with the ammonium magnesium phosphate. The addition of ferrous chloride will not prevent the precipitation of silica. It is recommended that the ammonium citrate solution should be added before the magnesia mixture, but if the two be added together the mixed reagent must be freshly prepared. Hydrogen peroxide may be used to oxidise any hydrogen sulphide present. W. P. S.

New Delicate Reaction for Elementary Oxygen. K. Binder and R. F. Weinland. (*Ber.*, 1913, **46**, 255-259.)—When an alkali is added to a mixture of pyrocatechol and a ferric salt, a salt of the complex tribasic acid $[Fe(C_6H_4O_2)_3]H_3$ is formed, having an intense dark red colour. When a ferrous salt is employed instead of the ferric, a pale red solution only is obtained which rapidly darkens with absorption of oxygen forming the ferric derivative. The complex ferrous salt is recommended as a delicate reagent for the detection and estimation of oxygen in gases. The reagent must be prepared with special precautions in the apparatus in which it is to be employed, in an atmosphere of purified hydrogen, freshly boiled water being used. The apparatus illustrated consists of a glass cylinder, *Z*, 21 cm. long and 2·6 cm. in diameter. A dropping funnel, *T*, of 100 c.c. capacity passes just through the cork, while a capillary tube, *K*, with tap *C*, goes almost down to the bottom of the cylinder. *V* is a gas-trap containing water.



0·4 grm. of purest ferrous ammonium sulphate and 0·5 grm. of pyrocatechol are placed in the dry cylinder. The stem and part of the bulb of the dropping funnel are filled with boiled water, and the rubber stopper carrying the funnel and capillary is inserted in the cylinder. Hydrogen gas, purified from oxygen by passing through two washing bottles containing an alkaline solution of sodium hydrosulphite, is then passed through the cylinder by way of the capillary, taps *C* and *A* being open. About 60 c.c. of boiled water and 5 drops of dilute sulphuric acid are placed in the funnel *T*, and purified hydrogen is passed in through the tube *R*, reaching nearly to the bottom of the funnel. The passage of hydrogen is continued for about fifteen minutes, the tap *D* is then closed and the tap *C* nearly closed. The water is next admitted to the cylinder, but the tap *B* is closed before the funnel is quite empty. The current of gas through the capillary is increased by opening the tap *C* again, about 15 c.c. of 15 per cent. potassium hydroxide solution are placed in the funnel and treated with hydrogen by opening the tap *D*. After passage of the gas for about forty-five minutes, the alkali is admitted to the reagents in the cylinder and all the taps are closed. The reagent should then be nearly colourless. The capillary tube *K* is filled above the tap with water freed from air by the passage of hydrogen and then connected with the source of gas to be tested; the latter is passed through the reagent in a slow stream regulated by the tap *A*, and if oxygen be present the liquid becomes coloured red. If the tap *C* be provided with a 3-way bore, the introduction of the sample and the displacement of air from the leading tubes may be simplified. The test may be made quantitative by preparing standard solutions of the complex ferric salt, diluting them with weak alkali and matching them colorimetrically with the reagent after the passage of a measured volume of gas. In ordinary gas analysis the ferrous reagent may be used for the absorption of oxygen in the Hempel pipette. A solution of 14 grms. of ferrous sulphate and 18 grms. of pyrocatechol in 75 c.c. of acidulated water is introduced into the pipette, cooled with ice, and a solution of 33 grms. of potassium hydroxide in 75 c.c. of water is then added. For the quantitative absorption of oxygen from a sample of gas, the gas must be shaken with the reagent in the pipette for five minutes. J. F. B.

Estimation of Platinum by Precipitation as Sulphide. R. Gaze. (*Apoth. Zeit.*, 1912, **27**, 959-960; through *Chem. Zentralbl.*, 1913, I., 464.)—Murmann has shown (ANALYST, 1899, **24**, 51) that the addition of mercuric chloride to zinc salts before the precipitation of the zinc as sulphide enables the zinc sulphide to be filtered without difficulty. In the case of platinum, a similar addition of mercuric chloride is of advantage; the quantity of mercuric chloride added should be from 5 to 10 per cent. of the weight of the platinum, and if it is desired to estimate chlorine in the same portion of the solution as the platinum, mercuric nitrate may be used in place of the chloride. For the analysis of barium platini-cyanide, a portion of the salt is heated with sulphuric acid and then with *aqua regia* until the barium sulphate is white in colour; the excess of *aqua regia* is then evaporated, the residue is diluted with water, the mixture cooled, and the barium sulphate collected on a filter. The filtrate is then concentrated, mercuric chloride is added, and the platinum is precipitated with hydrogen sulphide. An alternative method consists in igniting the salt over the blow-pipe, moistening the residue with nitric acid, and evaporating to dryness. The platinum is then extracted from this residue with *aqua regia* and precipitated as described. W. P. S.

Purity of the International Radium Standard. E. Haschek and O. Hönigschmid. (*Monatsh.*, 1913, **34**, 351-357.)—Last year the Vienna radium standard was compared actinometrically with the international standard of radium chloride prepared by Mme. Curie. The two preparations agreed within the limits of error of the method employed, which is taken as about 1 in 300. Mme. Curie has not described the preparation of the international standard, but it is reasonable to suppose that she adopted the method described by her in 1907 in her communication on the atomic weight of radium. This method—repeated recrystallisation from dilute hydrochloric acid—was adopted by Hönigschmid in Vienna. In 1907 Mme.

Curie recorded the fact that her purest radium bromide contained spectroscopic traces of barium, though certainly not more than 0·06 per cent. As this amount would introduce an error of 6 units in the second decimal place of the atomic weight, the authors subjected the Vienna standard, on which Hönigschmid's value for the atomic weight of 225·95 was based, to stringent tests for barium. The metal could not be detected in the Vienna standard, but it was detected and its amount approximately estimated (spectroscopically) in the mother-liquors from the preparation of the standard. On the assumption that the standard preparation still contains as much barium as remains in the mother-liquors, the standard may contain 0·004 per cent., but not more, barium. The radium bromide used by the same author cannot have contained more than half this amount of barium. Such amounts of barium would only affect an atomic weight determination 2 or 4 units in the third decimal place, and the atomic weight of radium is not known within ±0·012 per cent. The authors express the view that the Paris standard is most probably of the same order of purity as the Vienna preparation; that for actinometric purposes, with existing methods of conducting measurements, both may be regarded as pure, but that, should methods of measurement be so far improved as to justify a correction, then they should be regarded as of only 99·996 per cent. purity. G. C. J.

Estimation of Iron in Water. E. Tassilly. (*Bull. Soc. Chim.*, 1913, **13**, 34-37.)—The method is based upon the fact that in solutions of ferric thiocyanate containing a large excess of potassium thiocyanate the degree of spectroscopic absorption is proportional to the quantity of iron present. One hundred c.c. of the water are boiled with 20 c.c. of hydrochloric acid, 0·5 to 1 grm. of potassium chlorate added, and the boiling continued until chlorine ceases to be evolved. After cooling, 20 c.c. of potassium thiocyanate solution (17 grms. per litre) are added, and the liquid made up to 100 c.c. and examined in Féry's spectrophotometer in a 2 c.c. trough, the absorption due to the water being compensated by the use of a second trough containing water. The proportion of iron is then found from the reading by reference to the curves already established by Féry. The effect of nitrates is negligible, whilst that of sulphates is reduced to a minimum in the presence of hydrochloric acid. C. A. M.

Determination of Moisture. G. N. Huntly and J. H. Coste. (*J. Soc. Chem. Ind.*, 1913, **32**, 62-67.)—The Eighth International Congress of Applied Chemistry appointed a sub-committee to deal with the determination of water in coal, and their findings were published in the general report of the International Analysis Committee, and reprinted in the Transactions of the Congress. The present paper is mainly based on this report, but in addition deals with the determination of moisture by all methods likely to be of use in the drying of materials which through oxidation in air, or for other reasons, present difficulties.

(A) *Direct Methods.*—1. Water driven off by ignition, condensed in part of ignition tube and weighed directly (Penfield). Specially suitable for the analysis of rocks, giving accurate results in presence of carbonates or ferrous salts, but cannot be applied to organic substances.

2. Substance heated in a current of dry gas or in a vacuum, water vapour collected in calcium chloride or sulphuric acid and weighed, an accurate and widely applicable method; but air is not a safe gas to use, as with coal at 100° C. oxidation occurs. The use of inert gases, however, lessens the practical value of the process; and in the case of coal at any rate, coal-gas, unless carefully purified, would seem to be risky, since it contains gases absorbed by coal.

3. Substance mixed with an excess of a volatile, non-miscible liquid such as xylene, distilled, and the water under the hydrocarbon layer measured, with or without corrections for the mutual solubilities of the water and liquid used for its removal. A promising method and deserving of careful comparative study against Nos. 2 or 8, especially for difficult substances such as oils, butter, tar, and coal. Constam and Allen and Jacobs both describe the results of experiments in which this method is employed for the estimation of water in oils, fats, soaps, resins, coal, and petroleum. The method has the drawback of requiring close attention during the distillation, and is not free from danger of fire.

4. The material directly heated to 130° C. by a vapour jacket (high pressure steam), the steam given off condensed, and measured. A method applicable for dealing with samples of coals weighing as much as 1 lb. and has the advantage of requiring only a rough balance. It does not require skilled handling, and is eminently suitable for use in a power-station where results are required immediately. Allen and Jacobs distill 200 grms. of petroleum in the ordinary way, and then separate and weigh the water. The last method is exact to within 0·03 per cent.

(B) *Gasometric Methods.*—5. The substance is mixed with calcium carbide; acetylene measured.

6. The substance is mixed with magnesium methyl halide in presence of a suitable dry solvent, and the methane measured.

7. The substance is treated with sodium and the hydrogen measured. The carbide method has been applied to cordite, wool, butter, cocoa, coffee, and coal (see following abstract). Treatment with sodium has been used, as well as treatment with calcium carbide, by Allen and Jacobs for the determination of water in petroleum and its products, in which case the method is very accurate, since hydrogen is insoluble in oil; 2 grms. of finely cut sodium are used for each grm. of water supposed to be present, and the apparatus employed is similar to that for the carbide method.

(C) *Indirect Methods.*—8. Determination of the loss of weight by heating to a definite or indefinite temperature. The usual method. Correct results will be obtained if (*a*) the temperature of the oven and period of drying is sufficient to drive off all the moisture; (*b*) no moisture is reabsorbed before weighing; (*c*) no volatile substance other than water vapour is evolved; (*d*) no oxidation takes place; (*e*) no absorption of carbon dioxide takes place; (*f*) no internal chemical change is induced which may lead to the formation of water in the substance to be dried. All these points are discussed at some length, and the experience of different members of the sub-committee are summarised, considerable space being given to this section owing to the universality of the employment of the ordinary moisture oven.

9. Prolonged exposure in a vacuum in presence of strong sulphuric acid, either at

ordinary or at higher temperatures. It is important that the vacuum should be at least under 2 mm. (a pump of the Fleuss type will easily get the pressure down to 1 mm.). Before removing the samples from the exhausted desiccator dry air should be admitted. The sulphuric acid should be concentrated (1·84) and have a large effective surface. Figures are given for the loss of weight in a number of coals as found by different observers, and it is probable that by working with a really high vacuum (0·01 mm.), such as can be obtained by a double oil pump working in series, the time required for a vacuum moisture determination might be reduced to one or two hours. The paper concludes with a list of recommendations and conclusions arrived at by the International Reporters, which refer more particularly to coal, but, as this presents most of the difficulties which are found with other substances, most of the conclusions may be considered as of general application.

It is improbable, the authors say, that either the direct or the vacuum method will be adopted to any great extent, notwithstanding their undoubted merits; moreover, in various statutory rules and regulations (*e.g.*, the Fertilizers and Feeding Stuffs Act, 1906), it is provided that water shall be determined by drying at 100° C. For general work, temperatures below 100° C. are not nearly as satisfactory as those a few degrees above, both on the grounds of speed and accuracy.

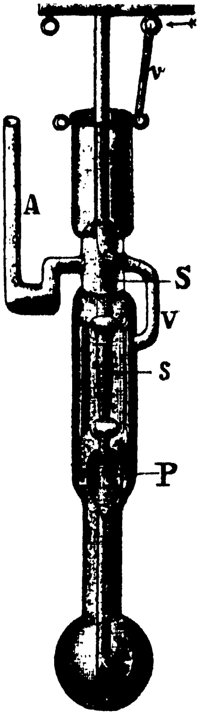
The Committee recommend that (1) whenever possible, especially in the case of very moist coals, the water in the original sample should be determined after it has been subjected to a minimum amount of preliminary mechanical treatment by either of the following methods: (*a*) A carefully weighed portion of from 100 to 500 grms. of coal is spread out on a metal tray and exposed for at least twenty-four hours to the atmosphere of the sampling-room. The tray containing the coal is then re-weighed and the percentage change of weight calculated. The coal is then coarsely ground (a ball-grinder is best) and a water determination concluded by one of the methods given in Recommendations 2, 3, or 4. (*b*) The xylene method used by Constam (Recommendation 5). (2) For ordinary technical work coal should be dried in an oven with a good air circulation for a period not exceeding one hour at a temperature not below 104° nor above 111° C. This temperature can be obtained by the vapour of boiling toluene, by steam at a pressure of 906 to 1,100 mm., or by an electrically heated thermostatic arrangement. An oven, such as described by Siau (ANALYST, 1911, **36**, 126) or Coste (*ibid.*, 1912, **37**, 385), is suitable. Air-ovens, and all devices in which the temperature of the walls is variable, are strongly condemned. A pre-heated supply of air is to be desired, and mere heating without a proper air-current is unreliable. (3) When it is suspected that a coal is specially liable to oxidation, or the half-hour and one-hour results differ materially, drying at 104° to 111° C. in a current of nitrogen, or carbon dioxide is to be preferred to drying in an oven through which a current of air is passing. The method of drying in a current of inert gas which must be quite free from oxygen is to be preferred for all exact work. (4) Drying to constant or minimum weight in a vacuum in the presence of sulphuric acid possesses no advantage over the last-mentioned method as it is much slower. Unless the vacuum is very high, it is better to fill the desiccator with carbon dioxide before exhausting. The coal should be weighed in watch-glasses, and the loss after 24, 72 and 144 hours should be determined, when the results are practically identical with those by method 3. (5) The xylene method is worthy of serious consideration, though in view of the limited information at the Committee's disposal it cannot be recommended unconditionally. H. F. E. H.

Determination of Moisture in Organic Substances. F. H. Campbell. (*J. Soc. Chem, Ind.*, 1913, **32**, 67-70.)—The method involves the use of calcium carbide in a new form of apparatus, and is claimed to be as accurate as the cold vacuum method, and more rapid than any of the recognised methods, while the necessary apparatus is both simple and inexpensive. The necessity for its use only arises when the material gives low results owing to oxidation when heated in air (*cf.* ANALYST, 1911, **36**, 112). The principle adopted is that of Rivett (*ibid.*, 1912, **37**, 51) in which the acetylene evolved is determined gravimetrically by difference, and the factor obtained (1 grm. acetylene = 1·44 grms. water) agrees closely with that found to hold by Rivett (*loc. cit.*). The apparatus consists of a tube about 12 cms. long and 1·5 cms. in diameter bent half-way at an angle of 45°, and closed at the lower end. Into the upper open end is fitted another tube holding the carbide and fitted with a stopcock leading to a washing bottle containing strong sulphuric acid. Calcium carbide is shaken down into the lower tube which contains the material to be tested, and the liberated acetylene escapes through the tube packed with carbide, being thus dried in its passage. Air is finally aspirated through the apparatus, which is heated towards the end of the gas evolution, and the loss of weight is a measure of the acetylene evolved. The results of a large number of comparative determinations are recorded in which roasted coffee, sweetened cocoa, tea, butter, and coals are dried by various methods. The time required for a determination varies from thirty to sixty minutes, and from 1 to 2 grms. of material as a rule are used. H. F. E. H.

Determination of Moisture in Foods and other Organic Substances. W. P. Skertchley. (*J. Soc. Chem. Ind.*, 1913, **32**, 70.)—A comparison is made of the loss of water from a number of materials when dried: (*a*) *In vacuo* for about twenty-four hours, and (*b*) in a water-oven at 100° C. for two hours. The substances generally yielded more moisture when dried *in vacuo*, especially in the case of farinaceous materials, which, when heated for more than two hours, began to increase in weight to the extent of a few mgrms. for each period of one hour. Working on a sample of biscuit the moisture apparently fell from 6·92 per cent. in two hours to 6·6 per cent. after six hours when dried in a water-oven at 100° C., whereas the moisture value rose in one case from 7·39 to 7·65 per cent., and in another from 7·13 to 7·48 per cent. when the drying-time was increased from twenty-four hours to forty-eight hours *in vacuo*. H. F. E. H.

Estimation of Water in Vegetables by Hoffmann's Apparatus. St. von Haydin. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1913, **25**, 158-160.)—In this process the substance is heated in a copper flask with ordinary petroleum (*paraffinum liquidum*) and oil of turpentine, the distillate being collected in a narrow graduated vessel, and the water measured under the hydrocarbon layer. It is recommended that 2 grms. of the finely divided vegetable be placed in a wire-gauze cage, which is then immersed in the petroleum in the copper flask; 20 c.c. of oil of turpentine are added, and the contents of the flask are heated to 180° C. A further quantity of 20 c.c. of oil of turpentine are then added through a tapped funnel, and the temperature raised to 200° C. About five minutes' heating at this temperature is sufficient to remove the last traces of water from the flask, and the whole operation requires only twenty minutes. The results obtained are concordant and agree with the figures found on drying the substance to constant weight in a water-oven. (See also ANALYST, 1907, **32**, 21; 1908, **33**, 97.) W. P. S.

APPARATUS, ETC.



Multiviscosimeter. I. Kurzmann. (*Chem. Zeit.*, 1913, **37**, 234.)—The multiviscosimeter, which is of the enclosed type, is fitted with a series of interchangeable capillaries, so that one instrument covers a large range of viscosities. The liquid under examination is measured into a bulb at the bottom, and forced up by air admitted through the stopper above, acting as a 3-way cock, into the capillary pipette, which is supported by the stopper in the axis of the instrument. The air displaced from the pipette reaches the atmosphere through a side U-tube which may contain some of the sample, so that the liquid is protected from alteration by evaporation. When the stopper is turned through 180 degrees the pressure on the two surfaces of the liquid is equalised, and the flow from the pipette takes place under the influence of gravity alone. Results at various temperatures agreed satisfactorily with those obtained with an Ostwald viscosimeter. The instrument is made by Aloys Schmidts Glasbläserei, Breslau I. O. E. M.

Portable Pettersson - Palmqvist Apparatus. R. P. Anderson. (*J. Amer. Chem. Soc.*, 1913, **35**, 162-168.)—The apparatus described and figured in the paper differs from earlier forms mainly in the dimensions of the gas burette, the principal portion of which is only about three times as long as it is broad, the graduated small bore portion lying nearly horizontally. One result of this arrangement is that the whole apparatus can be accommodated in a comparatively small rectangular glass cell, the outside dimensions of the case being 11 × 18 × 42 cm., as compared with 24 × 24 × 92 cm., which were the dimensions of the apparatus formerly made by Greiner and Friedrichs. There is also a great saving in weight. Another novel feature is a convoluted copper tube lying wholly within the water-bath, and serving to convey the gas samples or air from dwelling rooms to the burette, and effectually cool it to the temperature of the bath, thus making it possible to draw gas from a hot source, and proceed with its analysis without any delay. The apparatus is made by Greiner and Friedrichs, Stützerbach in Thüringen. G. C. J.

Small Apparatus for Electrochemical Analysis. F. Chancel. (*Bull. Soc. Chim.*, 1913, **13**, 74-76.)—The cell is a Jena glass test-tube 30 mm. in diameter and 125 mm. high. The anode is a platiniridium wire, 1 mm. in diameter and 50 mm. long, which is sealed through the centre of the bottom of the test-tube so that its top stands about 40 cm. above the bottom of the tube. The cathode is a cylinder of platinum gauze, 20 mm. in diameter and 30 mm. high, supported by a wire 1 mm. in diameter and 110 to 120 mm. long, and weighing from 6 to 9 grms. With such apparatus, a current of 1·5 ampères and a temperature of 80° to 85° C., 0·3 grm. of copper can be deposited quantitatively from 30 c.c. of solution in forty-five minutes provided only a little nitric acid is present in addition to 2 c.c. of sulphuric acid. An equal weight of nickel can be deposited quantitatively and in the same time from an ammoniacal solution of the sulphate containing ammonium sulphate with a current of 1 ampère at 75° to 85° C. In presence of nitric acid the results are equally exact, but the time required for complete deposition is prolonged to several hours. G. C. J.

Colorimetric Methods for Water Analysis by the Use of Autenreith-Koenigsberger's Colorimeter. W. Autenreith and A. Funk. (*Zeitsch. anal. Chem.*, 1913, **52**, 137-167.)—Details of processes are given for the estimation of ammonia, nitrous acid, nitric acid, iron, lead, hydrogen sulphide, etc., in water by means of a colorimeter devised by the authors. The comparisons are made by the use of a Helmholz double plate, and the two vessels containing the test solution and the comparison solution respectively have flat sides, and are placed at right angles to one another. The vessel containing the comparison solution is wedge-shaped, and may be moved up or down relative to the other vessel, so that the same depth of colour may be obtained in both vessels. W. P. S.

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REPORT.

Western Australia. The Health Act, 1911: Food Standards and Regulations. (Published as a Supplement to the *Government Gazette*, November 1, 1912.)—The Regulations come into operation on May 1, 1913, except that relating to whisky, which comes into force on July 1, 1913, and provide for the proper labelling and description of foods, condiments, wines, oils, drugs, etc., which are sold in Western Australia. "Standards" are given in almost all cases, the maximum quantity of preservative allowable in foods, etc., is mentioned, and a list is given of the colouring matters which may be employed in certain cases. The methods of analysis and examination which must be followed in ascertaining the composition, quantity, or conformity, or want of conformity, to standard of any food or drug, are given in a Schedule to the Regulations. W. P. S.

REVIEWS.

NOTES ON CHEMICAL RESEARCH. By W. P. DREAPER. London: J. and A. Churchill. 1913. Pp. 68. Price 2s. 6d. net.

The ostensible object of this book is to guide a beginner in the paths of research, but the material offered in its pages will not modify the views of those who hold that the only useful form of early education in methods of scientific inquiry is personal association in the laboratory with a sympathetic and scientific inquirer. To such it must seem that a textbook of research is about as profitable as a correspondence class in skating. What benefit, for example, will a student gain from the following typical paragraph? "Speculations as to ultimate conditions must have no adverse influence on the value of the experimental method in the student's mind. Rather should the contrary effect be produced. The programme before the chemist is sufficiently definite. Investigation must be continued from the position it occupies to-day, extended in every possible direction by the direct aid of experiment, and carried to its utmost limit." It is not only pedantic but unhistorical to declare that "the beginner must guard against the conclusion that startling discoveries result from a casual observation on the part of some investigator. This is not so." Shades of Bessemer, Pasteur, Griess, and Perkin! The history of chemistry, like the history of conquest, bristles with discoveries of this order, and therein lies the romance, the lure, the zest. In writing "it has been generally agreed that no scientific discovery can with any justice be considered as due to accident," the author must attach his own special interpretation to the words "accident" and "discovery"; because the common meaning of "discovery" is the act of finding out, that of "accident" an unforeseen or unexpected event. The human element continues to be the cardinal factor in effort of any kind, from racing to research, and a young chemist who is ponderously informed that he cannot make a discovery until he has mastered accumulated experience in the branch he has chosen is not likely to discover anything, ever paralysed by the gloomy notion that all the discoveries have been made. If alluring hope of accidental discovery is to be abandoned, research will degenerate into a leaden-footed procession in search of the foreseen, in anticipation of the expected.

"In experimental investigation great attention must be paid to detail," rightly affirms the author; but it would not appear that the same recommendation applies to writing books, for the small volume is relieved by many mistakes which are not always typographical. The statement that Lavoisier "exploded the philogeston theory" brings its own reward, whilst a reference to "almost virgin ground" invites the piquant, if delicate, question whether virginity can be qualified, but the seven errors on p. 42 are merely uninteresting; moreover, it is annoying to find *régimé* (*sic*) printed in italics when this distinction is denied to "résumés" (also *sic*), because *résumé* dwells outside the English dictionary, while "regime" has passed into it.

M. O. FORSTER.

CERAMIC CHEMISTRY. By H. H. STEPHENSON. London: Davis Bros. 1912. Pp. 91. Price 6s.

This work hardly fulfils the promise of its title. Whilst it contains a large quantity of data as to the mixtures used in the ceramic industries, there is little attempt at a discussion of the chemistry of the processes, and the information given falls far short of the present state of chemical knowledge in respect to silicates. This is unfortunate, as the author's close practical knowledge of these industries is evident, and the avowed aim of the book, the advancement of the higher study of the subject in English Universities, is an excellent one. Physical chemistry has been applied to the investigation of silicates by many able workers, with remarkable results, and the subject deserves more attention than it has received from chemists, outside of those engaged in the study of chemical mineralogy.

The notes on the analysis of clays are very scanty, and an authoritative account of the most trustworthy analytical methods would be welcomed by many analysts. Methods of proximate chemical analysis are discussed, and their errors indicated, but more might have been said of the microscopical method, whilst the important thermal method of study, so successfully employed in several laboratories at the present day, does not receive mention.

There is much information of interest to the chemist in the chapters on bodies, glazes and enamels, and the reader derives a very definite impression of the vast field for research in these directions which still remains open.

The author adopts the attitude that plumbism may be prevented by the adoption of fritted lead glazes, and the official Home Office test for solubility is described and approved. C. H. DESCH.

MATÉRIAUX DE GROS-OUVRE. Par E. LEDUC and G. CHENU. Paris and Liége: C. H. Bérangeur. 1913. Pp. 285. Price 6 francs.

This little volume, which is one of a series of manuals on branches of applied analytical chemistry under the general authorship of MM. Borda and Roux, deals with the properties and methods of examination of natural and artificial building stones, bricks and tiles, slates, paving materials, paints, clays, ceramic products, and glasses. Many analyses of such materials are given, and although most of the examples are French, which thus reduces their interest for English readers, one feels that information of this kind is much required. It is, at any rate, difficult to find when wanted.

It can hardly be expected that so small a book should give full accounts of analytical methods suitable for all the materials treated, but it is surely unnecessary, even in the interests of brevity, to advise fusion with alkaline carbonates for the decomposition of limestone which can readily be dissolved in dilute hydrochloric acid. The description which is otherwise good is of a method to cover all building-stones including granite, hence limestone is treated as a silicate.

Analysts who expend ingenuity in devising new "CO_2 apparatus" would do well to consider these words: "Pour doser l'acide carbonique il est necessaire d'employer la méthode classique par absorption et pesée, car les appareils multiples imaginés pour l'exécution de ce dosage, donnent des resultats trop éloignés de la vérité." The use of calcium chloride—a very poor desiccant for drying carbon dioxide, even when it is stated that the chloride should be neutral—is very undesirable. The

U-tubes shown in the figure of the apparatus to be used for this determination are of an early pattern with very large corks.

The superior utility of physical tests to chemical analysis is, in many cases with reason, frequently insisted upon.

Some interesting information as to comparative tests with Seger cones and pyrometers (apparently a Le Chatelier thermo-couple) does not tend to strengthen a somewhat weak faith in the accuracy of the cones.

The book is well illustrated with figures of testing machinery and apparatus, and of materials which have suffered under testing. It should be useful to many workers, not so much as an analytical manual, but by reason of the information as to what to look for and what properties to determine in the materials described. Its utility is somewhat reduced by the lack of an index. J. H. Coste.

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INSTITUTE OF CHEMISTRY.

The Thirty-fifth Annual General Meeting of the Institute of Chemistry was held at 30, Bloomsbury Square, on Monday, March 3, Professor Raphael Meldola, D.Sc., F.R.S., President, in the chair.

After the routine business had been transacted the President, Professor Meldola, delivered an address. He remarked that the past year had been one of great activity, and that the Institute was to be congratulated upon the substantial progress made in several directions. In thanking the members for his election to the chair, he said that he was glad to have an opportunity of making a confession of a change of view concerning the function of the Institute. At the time of its foundation he was one of those who had felt that no justification could be found for the creation of another corporation of chemists, but the more he had seen of its work the more strongly had he become convinced that the Institute had a public mission to fulfil of precisely the same order of importance as that discharged by any other body of professional men who serve the community. The applications of chemistry in every field of human activity had been steadily increasing, and the importance of professional chemists to the public welfare was becoming more and more recognised. They had not secured that full measure of public recognition to which they were entitled, but in this country all scientific affairs moved but slowly. The consolidation and the elevation of the profession and the maintenance of the status of the chemical practitioner would become more and more determined in the future by the standard of efficiency and of conduct set up by the Fellows and Associates. The most important advance made in the year was the adoption by the Fellows and Associates, at an Extraordinary General Meeting convened for that purpose, of a scheme for securing a site and erecting a building for new headquarters for the Institute. This had been rendered possible by the raising of a fund, which, although not complete, was considered sufficient to warrant the acquisition of the site. It had been originally estimated that about £15,000 would be required. Of this sum over £11,250 had been promised, £9,000 having been actually received. As far as could be gathered, it was probable that a sum of at least £16,000 would be required, so that an endeavour had yet to be made to raise a further £5,000. The completion of the

scheme cannot but be the most earnest wish of all who have at heart the reputation of this country, whose individual workers have contributed so nobly to the advancement of chemical science. It was hoped to create an establishment worthy of the dignity of the Institute as a professional body, and of the service rendered to the community by its members. Mr. John J. Burnet had been appointed as architect, and it was expected that building operations would begin in May. The President then mentioned the names of Fellows who had been specially helpful in raising the Buildings Fund, and also the names of the great City companies, manufacturing firms, and others, which were to be found in the list of contributors. Some of the companies had expressed their pleasure in having an opportunity of subscribing, for the reason that they had received so much advantage from chemical control in their industry, and other similar messages of sympathy and encouragement had been received.

Continuing, the President referred to the recognition accorded to the qualifications of the Institute by Government Departments at home, in the over-seas Dominions and in India. The Institute had taken its place as an imperial organisation, and it had set before it an ideal for which it was striving. It was hoped that in time every British chemist holding any position of responsibility, whether in the public service, in the teaching profession, in the chemical factory, or as a private practitioner, would be found on the register, and that the appearance of a name on that register should become a necessary public guarantee of efficiency in the same sense that a name on the Medical Register entitled a medical man to practise. It might be long before the whole of this ideal was reached, but the general movement was assuredly in the right direction. While the status of the Institute was recognised as a collective body, it could not be said that the recognition of the members as individuals was satisfactory. This was equally true of the public analyst, the teacher, and the chemical technologist. The question of remuneration was very much a matter of individuality, and depended on three factors—the place where the work was done, the actual value of the work to the employer, and the professional status and personal character of the employee. These factors were so variable that it would be as impossible to standardise the value of every particular member as it would be to enforce a particular scale of remuneration for each individual member of any other professional body. It was thought that it might be possible to draw up a schedule of fees for the more routine work, which would represent adequate remuneration, and a committee of the Institute was engaged in this task. If found practicable, the schedule when issued should at any rate serve as a useful guide to members in dealing with clients and public bodies. It might be impossible for the Institute to lay down compulsory regulations as to the fee to be charged, but it would perhaps engender a general understanding among the members which would make it distinctly known that the services rendered to the community by professional chemists in every department of their work had been very much under-estimated. Until the whole level of public appreciation of the value of this profession was raised, the country was destined to lose the services of that highest type of cultured and trained chemist of which other nations are more wisely availing themselves, to our detriment and their advantage. The Institute had sent representatives to give evidence concerning chemists in Government employment before the Royal Commission on the

Civil Service. The best thanks of the Institute were due to Sir William Tilden and Sir William Ramsay for their services in this connection, and it was earnestly hoped that some practical amelioration of the existing state of affairs would result. One very important step in the desired direction would be made if the Commissioners could only see their way to adopt the recommendation that Government Chemical Departments should be controlled by chemists. Turning to the question of the future development of the Institute, the President indicated the desire of the Council to arrange a Conference of Professors of Chemistry to consider the relationship of the qualifications of the Institute to those granted by other educational institutions. Was it not desirable, in the interests of the chemical profession in its broadest sense, that in an understanding with the teaching portion of the profession those who represent the collective body of practitioners should arrive at some concordant scheme of action which should be helpful alike to teaching institutions and to the Institute, and so to the cause of the chemical profession as a united profession in this country? An endeavour should be made to make the interests of the teachers and practitioners identical, and so avoid conflict, wasteful repetition, overlapping, and frittering away of resources, which had been the bane of educational development in this country. It was not suggested that the Institute should assume dictatorial powers. The situation had to be faced, and there were many problems which could only be solved by coming to an understanding with the educational authorities; and it certainly appeared that some effort towards standardising the chemical curricula of the country in relation to the examinations of the Institute should be made, or, at any rate, an opportunity given for an exchange of views among the teachers concerned.

A ballot having been taken, Dr. George Beilby, F.R.S., Dr. Frank Clowes, Professor P. F. Frankland, F.R.S., and Mr. David Howard were declared elected as censors.

The following were elected officers and Council:

President.—Raphael Meldola, D.Sc., LL.D., F.R.S.

Vice-Presidents.—George Thomas Beilby, LL.D., F.R.S.; George McGowan, Ph.D.; Sir Alexander Pedler, C.I.E., F.R.S.; Sir Boverton Redwood, Bart., D.Sc.; Sir William Augustus Tilden, D.Sc., LL.D., F.R.S.; Edward William Voelcker, A.R.S.M.

Hon. Treasurer.—Alfred Gordon Salamon, A.R.S.M.

Members of Council.—Leonard Archbutt; Francis William Frederick Arnaud; Edward John Bevan; Bertram Blount; Arthur George Bloxam; Cecil Howard Cribb, B.Sc.; William Salvador Curphey; Cyril Dickinson, B.Sc.; William Porter Dreaper; Martin Onslow Forster, D.Sc., F.R.S.; Sir Richard Garton; Frank William Harbord, A.R.S.M.; Arthur Harden, D.Sc., Ph.D., F.R.S.; Otto Hehner; Charles Alexander Hill, B.Sc.; Edward Hinks, B.Sc.; William Macnab; Gordon Wickham Monier-Williams, B.A., Ph.D.; William Henry Perkin, LL.D., Ph.D., F.R.S.; Thomas Slater Price, D.Sc., Ph.D.; Sylvester Oliffe Richmond; Clarence Arthur Seyler, B.Sc.; Thomas Stenhouse, Jr., B.Sc., A.R.S.M.; Frederick Wallis Stoddart; Oliver Trigger; James Woodward, B.A., B.Sc.; Sydney Young, D.Sc., F.R.S.

MAY, 1913. Vol. XXXVIII., No. 446

THE ANALYST.

OBITUARY.

JOHN HERON.

JOHN HERON was born in Bandon, co. Cork, on May 8, 1850. He studied engineering at Queen's College, Cork, and obtained the B.E. degree in 1871, but soon afterwards decided to make chemistry his career, and entered the Royal College of Chemistry under Professor Edward Frankland. In 1876 he left the college to take charge of a guncotton factory in Wales, but early in 1877 resigned that appointment to go to Worthington's Brewery at Burton as assistant chemist under Dr. Horace Brown. In 1883 he went as chemist to the Anglo-Bavarian Brewery at Shepton Mallet, and in 1885 was appointed chemist to the Invert Sugar Works of Messrs. Garton, Hill and Co. Here he remained till 1895, when he set up in London as brewers' chemist and consultant, remaining in practice till his death. He died on March 29, leaving a widow and a family of three sons and seven daughters.

Always keenly interested in chemical research, Heron contributed many valuable papers, mainly connected with the chemistry of brewing and of sugar, to various scientific societies. Of these, the first was the valuable research, made jointly with Horace Brown, "Contributions to the History of Starch and its Transformations," read before the Chemical Society. Of his others we can here only mention his important papers on the use of the polariscope in brewing practice and analysis, those on the manufacture and analysis of invert sugar, and his numerous researches in the chemistry of brewing, malting, and hops.

He was an original member of the Laboratory Club, which developed into the Institute of Brewing, and also of the Society of Chemical Industry, and filled many important offices in both these societies. A man of great ability and genial character, his death will be felt amongst a large circle of scientific and social friends.

L. T. THORNE.

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PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

THE monthly meeting of the Society was held on Wednesday evening, April 2, in the Chemical Society's Rooms, Burlington House. The President, Mr. L. Archbutt, F.I.C., occupied the chair.

The minutes of the previous ordinary meeting were read and confirmed.

Certificates of proposal for election to membership in favour of Messrs. H. F. E. Hulton, A.I.C., C. T. Kingzett, F.I.C., and A. J. Parker, were read for the second time; and a certificate in favour of Mr. Frank Sturdy Sinnatt, 321, Great Clowes Street, Higher Broughton, Manchester, Lecturer in Chemistry at the School of Technology, Manchester, Assistant Lecturer in Chemistry in the University of Manchester, was read for the first time.

Messrs. A. C. Bescoby, M.A., T. W. F. Clark, F. E. Day, B.Sc., A.I.C., and J. F. Tocher, B.Sc., F.I.C., were elected members of the Society.

The following papers were read: "The Moisture in some English, Colonial, and Foreign Butters during 1910-1912, with a Note on the Mitchell-Walker Moisture Test," by L. Gowing-Scopes; "Egyptian Butter and Samna," by S. H. Trimen; "Note on Samna," by H. Droop Richmond, F.I.C.; "On a Simple Test for Differentiating between Cocoa Butter and 'Green Butters,'" by Cecil Revis and E. Richards Bolton; and "The Correct Way to Use Glycerine-Jelly in Mounting Microscopical Objects," by L. W. Stansell, F.I.C.

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THE VALUE OF THE GUAIACUM TEST FOR BLOODSTAINS.

BY HERBERT S. SHREWSBURY, F.I.C.

(*Read at the Meeting, December* 4, 1912.)

ALTHOUGH Sutherland, in his monograph on bloodstains,* agrees with many other authorities in condemning the guaiacum test for bloodstains, much experience in the use of it fully emphasises its value when properly carried out. It should never be relied upon without the application of confirmatory tests, but should be regarded as a sorting test. The following details describe the method used by the author:

Preparation of the Solution.—About 1 grm. of the resin is washed three times with rectified spirit, and the residue is then shaken with 100 c.c. of the same solvent until a pale straw-coloured extract is obtained. The solution *must* be freshly prepared; when only twenty-four hours old it gives poor results, and when sixteen days old gave no reaction with a bloodstain a year old which reacted well with a freshly-prepared solution.

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* "Bloodstains," by Major W. D. Sutherland, p. 24. Baillière, Tindall, and Cox, 1907.

Application and Interpretation of the Test.—A small portion of the fabric to be tested is placed on a 4-cm. Dreverhoff ash-free filter, laid flat in a white flat-bottomed porcelain dish and moistened with two or three drops of distilled water. The surrounding filter is examined for any red stains which may be extracted; if the stain is on a weapon or other article, it is gently rubbed with the moistened filter. When the fabric is moistened, one or two drops of the guaiacum solution are added, and if no colour is produced within a few seconds, a drop or two of hydrogen peroxide (20 vols.) is added. If blood only is present, no coloration is produced after addition of the guaiacum solution alone, but a characteristic blue colour is produced within one second of the addition of the peroxide.

Blood never reacts with guaiacum alone, whereas some substances, such as the oxidases, do.

The *immediate* reaction with guaiacum in presence of peroxide is a most important condition, the neglect of which has largely caused Sutherland to condemn the test. The reaction must occur within the second, and later colorations, which invariably develop, are to be disregarded. Guaiacum solution and hydrogen peroxide alone, if left to evaporate on a clean glazed tile, will gradually develop a faint blue coloration. No case has so far been met with in which the test carried out as above has indicated blood, and further tests have failed to confirm its presence.

If a negative result is obtained, it is useless to search further unless the circumstances are exceptional.

Specific Nature of Test.—Sutherland (*loc. cit.*) quotes a list of thirty substances which all give the characteristic "colour," but the real question is, Do they give a blood positive reaction—viz.: (*a*) A red aqueous extract; (*b*) a blue coloration within one second after peroxide is added; and (*c*) no blue coloration without it? All the thirty substances mentioned by Sutherland (with the exception of quinone) have been examined by the test applied as already described, and are taken in Sutherland's order. A few preliminary tests with the reagents themselves may first be described.

The guaiacum solution alone spontaneously evaporated to a residue which remained white. Guaiacum solution and hydrogen peroxide developed a very faint violet colour in sixty seconds. This colour changed to a very faint blue, and remained constant in intensity. Distilled water, guaiacum solution, and hydrogen peroxide, gave the same very faint blue colour. The same reagents tried with human blood on cotton fabric, a bloodstain four months old, gave a vivid blue coloration in one second after the addition of hydrogen peroxide. The first ten substances gave an immediate vivid blue with guaiacum solution alone, and therefore gave a negative blood reaction.

The substances were, potassium permanganate, manganese dioxide, lead peroxide, chlorine, iodine, bromine, nitrous acid, bleaching-powder, ferric chloride, potassium ferro- and ferri-cyanides. These substances, being strong oxidising agents, react with guaiacum alone. With regard to the bleaching-powder, it should be mentioned that, of the three samples tested, two of them were old, and these two gave a perfect blood-reaction (exclusive of the aqueous red extract), guaiacum alone giving no colour. Gum acacia, gluten, and milk, gave negative results, milk giving a colour after several minutes. Raw potato gave a faint blue after two seconds with guaiacum alone, and a positive reaction on the addition of the peroxide. The substances reacting are doubtless enzymes, but it is possible that the vegetable enzymes lose the power of reacting with guaiacum during the drying of vegetable stains on clothes. Pus gave a negative reaction unless accompanied by blood. Ammonium chloride gave a positive reaction, and sodium chloride, sweat, and chromium salts, all negative reactions. Salt solution of 10 and 14 per cent. concentrations required, respectively,

No. of Experiment.	Description of—				
	Stain.	Washing.	Washed Stain.	Test.	Reaction.
1	Author's blood on cambric ; twenty days old.	Incompletely under tap ; air-dried.	Distinct pale yellow-orange brown ; resembled slightly scorched linen.	Moistened with water ; applied guaiacum solution, followed by hydrogen peroxide.	Immediate blue coloration.
2	As in Experiment 1.	As completely as possible under tap ; air-dried.	As above, but much paler.	As in Experiment 1.	As in Experiment 1.
3	Author's blood on cambric ; fresh.	Immediately after shedding ; three rinsings under tap ; air-dried.	Invisible ; very faint indication by transmitted light.	Soaked one hour in distilled water ; applied guaiacum solution, followed by hydrogen peroxide.	Immediate faint blue, rapidly developing to sky blue ; stains white against blue background of fabric, corpuscles having apparently been washed from stains into fabric.
4	As in Experiment 3.	Five minutes after shedding ; thorough washing under tap.	As in Experiment 3.	As in Experiment 3.	As in Experiment 3.
5 and 6	As in Experiments 1 and 2.	As in Experiments 1 and 2.	As in Experiments 1 and 2.	No moistening with distilled water ; guaiacum solution and hydrogen peroxide applied.	Immediate blue coloration.

five and three seconds for the development of the blue colour. A 16 per cent. solution gave a faint positive result. Sutherland's suggestion, that the presence of salt in sweat and urine vitiated the test, is thus groundless. Sweat required thirty seconds to give a faint colour.

Rust required two minutes, the colour even then being very faint, but salts of copper and many plant extracts gave positive results. As regards these last, it is to be remembered that the reaction is characteristic of enzymes; but fortunately in practice, for reasons already suggested, these do not usually interfere with the test. Decoctions of leather, flannel (both old and new), and some kinds of filter-paper, were all negative. Four varieties of leather were tried, including oil, bark, and chrome tannages. Eleven kinds of filter-paper were experimented with, the best of all being Dreverhoff's ashless 400. Wheat and maize flour contained enzymes, and gave no colour with guaiacum solutions alone, but gave the blue colour with hydrogen peroxide. Spring waters will react if they contain ferric chloride.

Distilled water containing copper is mentioned by Sutherland. One part of copper sulphate in 10,000 was found to be negative, while 1 in 1,000 and 1 in 100 were positive; but it is inconceivable that distilled water would contain these latter amounts.

Reactions with Washed Bloodstains.—The experiments tabulated on p. 188 show that the guaiacum test will reveal the presence of a bloodstain that has been thoroughly washed.

It is not probable that clothing could be thoroughly washed within five minutes of a murder, so that Experiment 4 indicates the probability of the detection of a washed bloodstain by the guaiacum reaction. There are few, if any, stains except blood which will react to guaiacum after thorough washing. With a washed garment, therefore, a positive guaiacum reaction is highly characteristic of the presence of blood.

The test does not, of course, indicate human blood; but the possibility of any other source is frequently excluded by other considerations, the very fact of clothing having been washed being suspicious in certain circumstances.

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DISCUSSION.

Dr. WILLCOX said that this paper was an interesting one, and put very fairly the position that the guaiacum test was entitled to hold. It was a very delicate test, but its delicacy depended upon the reagents being good—the guaiacum must be fresh and the peroxide pure and of the proper strength. As Mr. Shrewsbury had mentioned, it could only be regarded as a sorting test, not capable of proving with certainty that blood was present; but there was a very strong presumption as to the presence of blood if the reaction was given quickly, this being the most important point. Some of the fine distinctions mentioned in the paper did not always hold good in actual practice. In medico-legal work the articles to be examined were often in a very dirty condition, and themselves yielded coloured solutions, so that the only thing that really enabled one to decide was whether a blue colour was given quickly or not. He could not quite agree with Mr. Shrewsbury's observation that the reaction was not given quickly by any substance but blood. He had frequently found that in the case of boots which were very dirty, and perhaps covered at the bottom with a sort of manure, including pieces of straw, grass, etc., the guaiacum reaction was given by the deposit on the soles almost as quickly as in the case of blood, although probably no blood was present. He ought perhaps to say that he had almost given up using the guaiacum test for medico-legal work, because he had found the recently introduced benzidine test to be more delicate and reliable as a sorting test. The reagent was a saturated solution of benzidine in glacial acetic acid, diluted with four times its volume of 10 per cent. hydrogen peroxide. When this mixture was placed upon a bloodstain, a magnificent blue colour was produced, like that obtained in the guaiacum test, but deeper. The benzidine test had the advantage over the guaiacum test that the reagents were not so liable to go wrong, but caution was necessary in regard to one point—namely, that the hydrogen peroxide must be perfectly pure and must contain no free acid. Of course, as Mr. Shrewsbury had pointed out, the indications of such preliminary tests must be confirmed by spectroscopic examination, by obtaining hæmin crystals, and, if possible, by the application of the serum test to ascertain from what animal the blood came; but when the articles to be examined were numerous the utility of these preliminary sorting tests was obvious. It sometimes happened that, owing to the minuteness of the quantity of material available, only the guaiacum or benzidine tests could be applied, and in such cases the evidence must remain correspondingly doubtful, unless there were other circumstances to confirm the indications of the test.

* * * * *

THE BACTERIAL TESTING OF DISINFECTANTS: A PRACTICAL CRITICISM.

By C. T. KINGZETT, F.I.C., and R. C. WOODCOCK, F.I.C.

(*Read at the Meeting, March* 5, 1913.)

In our paper (*Pharm. J.*, 1910, **85**, 157) on "Bacteriological Testings of Certain Disinfectants, and the Results as affected by Varying Conditions" of time and temperature, we clearly demonstrated by experiments the fact that, while the Rideal-Walker test is serviceable for ascertaining the comparative germicidal values of *coal-tar disinfectants* under the particular conditions of that test, it is perfectly useless for determining the real *disinfectant* values of these and other preparations for practical application.

For example, if peroxide of hydrogen of so-called 10 volumes strength, and formaldehyde of about 40 per cent. strength, be severally examined under the ordinary conditions observed in conducting that test, it appears that they are both of insignificant value as disinfectants, whereas it is a matter of common knowledge and beyond dispute that for many applications they are, in fact, more valuable than pure phenol and some of those other coal-tar preparations which, being peculiarly amenable to the Rideal-Walker test, appear in the light of that test to be superior.

TABLE.

Name.	General Remarks.	Dilution required to kill *B. Typhosus* in an average of 7½ minutes at 16° to 18° C.	Coefficient calculated upon the original material, unless otherwise stated.
Boric acid ...	100 grms. of water at 20° C. dissolve 4 grms. of the acid	—	Nil
Chromic acid, CrO_3 (anhydride)	A saturated solution at 18° C. contains 62·45 per cent.	1 : 300	3
Hydriodic acid ...	—	1 : 350	3·5
Hydrochloric acid	—	1 : 800	8
Nitric acid ...	—	1 : 650	6·5
Sulphuric acid ...	—	1 : 750	7·5
Sulphuretted hydrogen	100 c.c. of water at 20° C. dissolve 0·4 grm.	—	Nil
Sulphurous acid	—	1 : 1,350	13·5
Bleaching powder	Available chlorine, 32·6 per cent. (A clear solution was used.)	1 : 6,000	60
Bromine	A solution was made in distilled water; it contained 3 per cent. of bromine. A saturated solution at 25° C. is said to contain 3·07 to 3·48 per cent. of bromine	1 : 5,000	50
Chlorine	A solution was made in distilled water; it contained 0·22 per cent. of chlorine. A saturated solution at 25° C. is said to contain 0·641 per cent. of chlorine	1 : 20,000	200
Iodine	A solution in distilled water was used containing 0·023 per cent. of iodine. A saturated solution at 25° C. is said to contain 0·0279 to 0·034 per cent. of iodine	1 : 16,000	160
Acetic acid ...	—	1 : 40	0·4
Benzoic acid ...	Using a solution known to contain 0·263 grm. per 100 c.c. (*i.e.*, saturated at 18° C.)	1 : 380	3·8
	In another experiment	—	Nil
Citric acid ...	Very soluble	1 : 4	0·04
Formic acid ...	—	1 : 150	1·5
Lactic acid ...	—	1 : 170	1·7
Picric acid ...	100 grms. water at 20° C. dissolve 1·11 grms.	1 : 500	5
Prussic acid ...	—	1 : 50	0·5
Pyrogallol ...	Very soluble	1 : 40	0·4
Salicylic acid ...	100 c.c. of water at 20° C. dissolve 0·18 grm.	—	Nil

Name.	General Remarks.	Dilution required to kill *B. Typhosus* in an average of 7½ minutes at 16° to 18° C.	Coefficient calculated upon the original material, unless otherwise stated.
Tannic acid	Very soluble	1 : 14	0·14
Tartaric acid	„ „	1 : 5	0·05
Borax crystals	100 grms. of water at 21·5° C. dissolve 2·8 grms. of the anhydrous salt	—	Nil
Copper sulphate crystals	100 grms. of water at 20° C. dissolve 20·7 grms. of the anhydrous salt. No sharp reading obtainable	Up to 1 in 30	Nil to 0·3
Ferric chloride	—	1 : 8	0·08
Ferrous sulphate crystals	100 grms. of water at 20° C. dissolve 26·4 grms. of the anhydrous salt	—	Nil
Mercuric chloride	No sharp reading could be obtained. Sulphuretted hydrogen water was added to the broth tubes	1 : 1,500 to 1 : 2,000	15 to 20
Potash alum	100 c.c. water at 15° C. dissolve 9·6 grms.	—	Nil
Potassium bichromate crystals	A saturated solution at 20° C. contains 12 per cent. of the salt	1 : 15	0·15
Potassium bisulphate	—	1 : 450	4·5
Potassium permanganate	—	1 : 6,000	60
Potassium permanganate	50 per cent.	1 : 1,500	15
Zinc sulphate	50 per cent.		
Sodium bisulphite	—	—	Nil
Sodium carbonate	—	—	Nil
Sodium fluoride	100 grms. of saturated solution at 18° C. contains 4·3 grms.	—	Nil
Zinc chloride	—	1 : 17	0·17
Zinc sulphate crystals	100 grms. of water at 15° C. dissolve 50·88 grms. of the anhydrous salt	—	Nil
Copper benzoate	Very slightly soluble in water	—	Nil
Copper formate	A saturated solution equals 1 in 8 of water	1 : 10	0·1
Lead acetate crystals	100 grms. of water at 25° C. dissolve 50 grms. of the anhydrous salt	—	Nil
Mercuric cyanide	100 c.c. of water dissolve 8 grms. at 15° C.	1 : 200	2
Ortho-oxalic acid ester of phenol	—	1 : 520	5·2
Quinine bisulphate	100 grms. of water at 25° C. dissolve 11·77 grms.	—	Nil
Sodium salicylate	Very soluble	1 : 5 to 1 : 10	0·05 to 0·1

Name.	General Remarks.	Dilution required to kill *B. Typhosus* in an average of 7½ minutes at 16° to 18° C.	Coefficient calculated upon the original material, unless otherwise stated.
Strychnine sulphate	Water dissolves 3·2 per cent.	—	Nil
Zinc benzoate ...	Very slightly soluble in water	—	Nil
Zinc boro-diortho-hydroxy-benzoate	Not very soluble in water ...	1 : 300	3
Zinc formate ...	1 part dissolves in 24 parts of water	—	Nil
Zinc hydrogen borodisalicylate ...	Fairly soluble in cold water	1 : 400	4
Zinc salicylate ...	Soluble to about 2 per cent. in cold water	—	Nil
Zinc sulphocarbolate	Soluble in 2 parts of water	1 : 2 to 1 : 5	0·02 to 0·05
Ammonia (NH_3)		1 : 50	0·5
Caustic soda ..		1 : 1,400	14
Lime, slaked	Tested directly in suspension	1 : 450	4·5
Lime-water (saturated)	100 grms. of water at 20° C. dissolve 0·125 grm. of CaO	—	Nil
Aniline	100 c.c. water dissolve 3·481 c.c. aniline	1 : 55	0·55
Nicotine	Soluble in water in all proportions ...	1 : 20	2·0
Pyridine	German Government quality distilling 50 per cent. at 140° C., 90 per cent. at 160° C.	1 : 38	0·38
Strychnine ...	Water dissolves 0·02 per cent.		Nil
Alcohol C_2H_5OH		1 : 3	0·03
β-naphthol ...	A solution containing 5 per cent. of soda and 5 per cent. of β-naphthol was used, the coefficient likely to be due to the soda only		
	Coefficient of the solution	1 : 56	0·56
	But coefficient calculated on the original β-naphthol	1 : 1,120	11·2
Camphor... ...	1 part dissolves in 700 parts of water	—	Nil
Chloral hydrate...	Very soluble	1 : 9	0·09
Chloroform ...	A saturated water solution was used; 100 c.c. of water at 17·4° C. contain 0·71 grm. of chloroform	—	Nil
Eugenol	Only slightly soluble in water ...	1 : 900	9
Formaldehyde (38·5 per cent.)		1 : 35	0·35

Name.	General Remarks.	Dilution required to kill *B. Typhosus* in an average of 7½ minutes at 16° to 18° C.	Coefficient calculated upon the original material, unless otherwise stated.
Guaiacol... ...	Soluble in 80 parts of cold water ...	1 : 200	
Hydrogen peroxide	10 vols.	1 : 4	0·04
Hydro-naphthol...	The same results were obtained as with β-naphthol above		
Iodoform ...	100 c.c. water at 25° C. dissolves 0·01 grm.		Nil
Menthol	Almost insoluble in water	—	Nil
Methylene blue (double zinc salt)	Soluble in about 30 parts water ...	—	Nil
Resorcinol ...	Very soluble	1 : 30	0·3
Sulphur, flowers of	Moistened with alcohol and an 8 per cent. solution of gelatin used instead of water to suspend it	—	Under 0·1
Thyme oil (red oil of origanum)	In making the test, the oil was mixed with water and well agitated	1 : 2,800	
Thymol	1 part dissolves in 1,500 parts of cold water	1 : 2,400	24

The solubilities of substances given above have been taken from various books of reference.

This criticism applies equally to the several modifications of the Rideal-Walker test as advocated by the *Lancet* and others.

Illustrative of this conclusion, our experiments showed that one coal-tar disinfectant, having a Rideal-Walker coefficient of 4, exhibits a higher value than another possessed of a Rideal-Walker 20 coefficient when examined by the Martin and Chick method of testing.

Since then we have determined the Rideal-Walker coefficients of a great number of chemical substances, all of which are known to be possessed of either oxidant, antiseptic, disinfectant, preservative, corrosive, or lethal properties, and the results fully confirm our earlier conclusions, and suffice to establish the perfectly unreliable character and extremely limited capacity of the Rideal-Walker test for ascertaining the real disinfectant value of sanitary preparations.

It will be observed that the halogens, chlorine, bromine, and iodine, in the pure state, have phenomenally high coefficients, while borax, boric, salicylic and benzoic acids, the sulphates of iron, copper, and zinc, acetate of lead, the chlorides of iron and zinc, and strychnine, have little or no value in the light of this test, an obviously erroneous condemnation of value which is also shared by lime-water, menthol, iodoform, nicotine, camphor, chloroform, sodium fluoride, sodium bisulphite, and quinine bisulphate.

Again, the powerful oxidising agent, chromic acid, according to this criterion, has but the comparatively small coefficient of 3. Nicotine, prussic acid, and strychnine—three of the most lethal agents known to science—would appear to be practically devoid of the power to kill the common typhoid bacillus, and even mercuric chloride, which has in the past been generally credited with the highest germicidal power, is made to appear of smaller value than many coal-tar preparations when tested as described.

Curious to relate, potassium permanganate, which is known to be a powerful oxidising agent, comes out with twenty times the value of chromic acid, while peroxide of hydrogen, which is a valuable disinfectant and oxidising agent, is made to appear possessed only of the paltry coefficient 0·04.

The case of benzoic acid is worthy of a little special attention, bearing in mind more particularly the extent to which it is employed as a food preservative. It is but slightly soluble in water, and, using a saturated solution in our investigation, it appeared in one experiment to be devoid of bactericidal value, whilst in a second test it gave a coefficient of 3·8. According to Rideal, it has a coefficient of 5, but how this was determined is not known by us. In any case, it is remarkable that on the one hand by the same test the same substance is made to have about four times the value of pure phenol as a germicide, while on the other it is made to appear valueless. What can be thought of a test that gives such divergent results?

It is also of some interest to note that according to the Rideal-Walker test as applied by us, sulphate of copper, which has always been regarded as a germicide of some value, and which is often used very successfully for the purification of water-supplies, is almost devoid of germicidal value.

The truth is that the Rideal-Walker test has been credited with the most exaggerated capability, while it ignores the existence of chemical actions and conditions—in short, that "total environment," which is of the highest importance if proper appraisement is to be made of so many different articles which are known to exercise their chemical, bactericidal, and other actions in so many diverse ways.

In the Rideal-Walker method the typhoid bacillus is generally employed as the test organism, but other organisms may, of course, be, and are often, used, and the results obtained, using these different varieties, vary very much; in other words, the ratio of the killing power between carbolic acid and the disinfectant—the subject of test—varies to an enormous extent. Other investigators have already pointed this out, as illustrated by the following quotation from "The Bacteriological Examination of Disinfectants," by W. Partridge (1907): "Most disinfectants show different coefficients when tested against different organisms. For example, a well-known disinfectant has the following carbolic acid coefficients for the organisms specified:

B. typhosus	11·0
Staphylococcus pyogenes aureus	9·3
B. pestis	34·0
B. tuberculosis	11·0
B. dysenteriæ	10·0
B. diphtheriæ	20·0
Vibrio choleræ	32·3
B. mallei	15·0"

That is to say, this particular disinfectant could be advertised as having a coefficiency of 9·3 or any of the other figures up to 34.

It is further to be noted that the relative coefficients of a number of disinfectants in respect of the same individual organisms vary throughout the series.

The Rideal-Walker test, then, stands condemned as a measure of value of disinfectants as a class, and the only gauge of utility of the various disinfectants available for public and sanitary employment is the extent to which, as shown by experience, they may be satisfactorily employed for the particular purposes for which they are advocated.

If for foods, etc., preservative action be required, benzoic acid, borax, boracic acid, salicylic acid, and so forth, are available.

If destructive action by oxidation be desired, peroxide of hydrogen, chromic acid, the manganates, permanganates, chlorine, etc., can be usefully employed.

If coagulative effect be desired, carbolic acid, alum, and many other substances may be selected.

If antiseptic action be the desired object, chloride of zinc, the sulphites and bisulphites, benzoic acid and the benzoates, will serve with others.

If sterilisation by fumigation be aimed at, formaldehyde and burning sulphur in association with aqueous vapour will fully answer requirements. As to the first-named of these two substances, we have shown in our previous investigation that although formaldehyde exhibits a nominal Rideal-Walker coefficient of only 0·35, it shows a superiority of germicidal value in comparison with phenol when sufficient time be allowed and at a higher temperature. For example, in one and a quarter hours, at 15° to 18° C., it has almost the same killing power over *B. typhosus* as carbolic acid, and in one and a quarter hours at 37° C., 1 part in 550 of the 37 to 39 per cent. solution kills as compared with 1 in 320 of carbolic acid; in other words, the coefficient of formaldehyde then becomes 1·8 as compared with carbolic acid coefficient 1.

As to burning sulphur, its utility is supported incidentally by our test, showing sulphurous acid to have the coefficiency of 13·5.

For purification of the air, antiseptic washings of wounds, etc., and general sanitary requirements, many other preparations are available.

It is worthy of observation that the Special Sanitary Commissioner of the *Lancet* (October 19, 1912), in his report concerning the International Congress of Hygiene and Demography, recently held at Washington, expressed the view that the chief difficulty experienced by the Congress was to the effect that "the immense majority of members did not in the least understand what these labours should have aimed at," and this would certainly appear to be the case in respect of at least one of the resolutions which was passed thereat in the following terms: "That the disinfectants used in different countries should be controlled by a simple bacteria test, capable of being easily effected, and that a Committee from this Congress confer with the Committee of the International Congress on Applied Chemistry to define such test." At any rate, this resolution has nothing but an abstract value, and even that is destroyed by the practical results of our investigation, as reported in this communication, in view of which it is inconceivable that any such test can be devised.

It may be noted that the coefficients of some of the substances referred to in this investigation have been previously determined by others ("A Dictionary of Applied Chemistry," Thorpe, vol. ii., 1912, p. 263), and the following table gives the results side by side with our own in those cases where they agree:

Name.	Thorpe's Dictionary.	Our Determination.
Absolute alcohol	Less than 0·1	0·03
Acetic acid	0·6	0·4
Boric acid	Less than 0·1	Nil
Formaldehyde	0·55	0·35
Lactic acid	1·8	1·7
Pyrogallol	0·22	0·4
Resorcinol	0·3	0·3
Zinc chloride	0·15	0·17

An important feature of our investigation is that all the determinations of coefficient value were made by the same investigators working in the same laboratory under absolutely identical conditions.

We have pleasure in expressing our acknowledgment of the practical services given to us in this investigation by our assistant, Mr. J. E. Brimley, B.Sc.

DISCUSSION.

The PRESIDENT having invited discussion—

Mr. W. PARTRIDGE said that, although the Rideal-Walker test had been subjected to a great deal of criticism, in his opinion it would still be found to be the most reliable for most cases. Since it was brought forward, however, a good deal more had been learned about *B. typhosus*, and particularly the fact that this organism varied greatly in its power to resist the action of disinfectants. Indeed, there were so many varieties of it that it was not surprising that, even in the hands of the same worker, different coefficients should be given by organisms from different sources. In view of this he had sometimes wondered whether it would not be better to use another organism. It might be noted that some of the substances included in the authors' present investigation were hardly of the nature of disinfectants. Iodoform, for instance, was a substance which it was perhaps hardly fair to test by the ordinary methods. It became active, only when actually in contact with the surface to which it was applied, and its action was no doubt due to the iodine liberated from it. The results recorded in the authors' table, however, appeared to indicate that it had no disinfectant action at all.

Mr. F. W. RICHARDSON said he had always found the great difficulty to be in regard to the nature of the bacterial culture used. *B. typhosus* varied a great deal in character, and, as Mr. Partridge had indicated, it was almost impossible to get concordant results unless one could use a perfectly stable organism. He himself should prefer to use *B. coli communis*, and he suggested that an attempt should be made to ascertain what culture medium was best calculated to insure concordant results when this organism was used for such tests.

Mr. CHASTON CHAPMAN asked whether the authors intended to draw any, and if so, what, distinction between antiseptics and preservatives. He noticed, for instance, that sulphites and bisulphites were not placed in the category of food preservatives, for which purpose they were, in fact, somewhat extensively used. If this was to be taken to mean that those substances had any marked bactericidal properties, he thought the authors were scarcely correct, as unless used in excessive proportions, they were only capable of acting as preservatives.

Mr. RICHMOND thought that the fact that there were so many varieties of *B. coli* would militate against the adoption of Mr. Richardson's suggestion.

Mr. J. GOLDING said that his colleague, Dr. Williams, was in favour of the use of dried fæces rather than cultures of *B. coli* or *B. typhosus* alone. Cultures of these organisms were not really very constant, and in using a material containing organic matter the conditions under which most preservatives and disinfectants were required to act would be more nearly approached.

The PRESIDENT said that he felt some hesitation in contributing to the discussion, since the subject was of such a special character, and one with which he had only occasionally to deal; but he should like to ask why raw sewage should not be used for the purpose of these tests? Whenever he had had to compare the action of one disinfectant against another, he had always used raw sewage, and the results obtained seemed to him to be just as good, from a comparative point of view, as were obtained when a specific organism was used.

Dr. RIDEAL said that at the Washington Congress there were two debates on the testing of disinfectants, one in connection with the International Congress of Applied Chemistry, and the other in connection with the International Congress of Hygiene and Demography. The discussion in the chemical congress resulted in the formation of a committee, including representatives of France, Germany and America, with himself as the English representative, their instructions being as stated by Mr. Kingzett and Mr. Woodcock. Amongst the tests that had already been devised were the Rideal-Walker test, the test devised at the Lister Institute, and also the test devised by Dr. Sims Woodhead (the *Lancet* Commissioner) and Mr. Ponder. The *Lancet* test, however, after the discussion that took place at the British Pharmaceutical Conference at Cambridge, had not been heard of any more. Work had since been done on the subject in America, and Dr. Anderson, the head of the Bureau of Hygiene at Washington, in conjunction with Dr. McClintick, had devised a new test which was a sort of compromise between the *Lancet* test and the Rideal-Walker test. The results of the application of this test to all the disinfectants on sale in America were published as a bulletin of the Bureau of Hygiene, and were under discussion just before the two International Congresses met. Unfortunately, however, the death of Dr. McClintick, the worker who was best acquainted with the technique of the test, prevented its full discussion. As to whether *B. typhosus* or *B. coli* should be used, the amount of experience now available was greater with *B. typhosus* than with *B. coli*, and he thought the variations were less in the case of *B. typhosus* than in the case of *B. coli*. It was certainly true that in the *Lancet* test, using the McConkey medium, it was easy to see whether the *B. coli* was alive or not, but *B. coli* was open to great objection on account of its being so common. Accidents with *B. coli* were not at all unlikely, and, seeing that the organism was subject to great variations in strain, he should certainly advocate the continuance of the use of *B. typhosus*. He thought the authors would agree that a bacterial test, after all, did not really indicate whether proper disinfection was taking place. For this it was necessary to supplement the bacterial test by determining the chemical change. In the course of the *Lancet* inquiry, some attempt was made to show that that test had a direct relation to the chemical composition of the disinfectant, and the formula $\frac{P-D}{3}$ = *Lancet* figure was put forward. But in his criticism of that suggestion he had shown that, while an infinite disinfecting power would be assigned to carbolic acid, that assigned to tribromophenol would be nil. He agreed, however, with the present authors that the oxidising power of the disinfectant must be taken into consideration—*i.e.*, not only the bactericidal effect, but the effect on the environment of the organism. He thought that the most remarkable fact brought out by the recent work on this subject was that, whichever test was used, the halogens invariably showed a very high coefficient, chlorine being, according to all the methods, the pre-eminent bactericide. The utility of bleaching powder and the hypochlorites in the sterilisation of water was now recognised, especially in America, where in many large cities the use of these agents was solely relied upon for maintaining the purity of the water-supply, while the use of bleaching powder, or of chlorine itself, for sterilising sewage effluents before their discharge into rivers, was also very extensive in America; so that the indications given by these bacterial tests as to the germicidal value of chlorine compounds had been fully borne out by the results of practical application on a large scale.

Mr. E. K. **Rideal** said that König had shown that the acids, when subjected to a disinfecting test, showed coefficients varying according to their degree of ionisation, etc., thus bearing out the observations of the authors. The real question in these cases was whether the reaction was that of the substance itself or of its ions. In the case of phenol, the substance itself reacted, probably forming an emulsion; but in the case of, say, salicylic acid, which became partly ionised in solution, it did not seem right to assume that the salicyl ion would act upon an organism in the same manner as phenol, which was not necessarily ionised in solution, and this was no doubt the reason for the anomalous values shown by salicylic acid and benzoic acid and other compounds containing ions in solution. Substances which formed emulsions could no doubt be tested quite well by means of dried fæces, but substances that were ionised in solution would be at a disadvantage, as the ions would be discharged on coming in contact with the small suspended particles contained in such a medium. The Rideal-Walker test itself did not treat several oxidising agents quite fairly, because many oxidising agents spontaneously discharge their ions, even in the absence of organic matter. A saturated solution of ozone, for instance, would be decomposed when it came into contact with glass beads, or even with the sides of the vessel itself. If ozone were brought into contact with micro-organisms, two actions would take place, the organism being destroyed to a certain extent by the ozone, while at the same time on the surface of the organism the ozone would be catalytically decomposed. So that two distinct methods of testing were really needed, the one against an inorganic colloid and the other against the living organism itself, the two factors—namely, the ionic reaction and the coagulant (or rather surface tension) reaction—being made use of.

Mr. PARTRIDGE desired to add that, when he suggested the use of an organism other than *B. typhosus*, he was not thinking of *B. coli*. The liability of the latter to variations was well known, and he thought that the first step towards the improvement of bacterial tests should be to ascertain what organism was least likely to vary. Both *B. typhosus* and *B. coli* did vary, and very often to an extent sufficient to upset the results.

Mr. WOODCOCK said that iodoform had been included because it was used extensively as a surgical dressing, its action being at any rate antiseptic; and the table, of course, included other substances which had antiseptic properties, but which were not generally used as disinfectants. He did not think that the use of *B. typhosus* afforded any ground for complaint, provided that the growth was satisfactory—and if anything were wrong in this respect it would be at once detected by anyone experienced in the use of the test. But special experience in the details of manipulation was of course essential. With regard to the use of the terms "antiseptic" and "disinfectant," they had used the term "antiseptic" as denoting not actual killing power, but merely the restraining of growth or prevention of decomposition. They regarded "antiseptic" and "preservative" as having practically the same meaning.

Mr. KINGZETT said that there was no intention on their part merely to attack the Rideal-Walker test. They agreed with Mr. Partridge that there was a great deal to be said in its favour up to a point, and there was no doubt as to its being the best test yet available for determining the relative value of coal-tar disinfectants. But they did say that it was of little value in the case of preparations outside that class, because, owing to questions of environment, the substances were not really amenable to the test, seeing that no regard was had to chemical and physical conditions, which, as Mr. E. K. Rideal had pointed out, were of so much importance in considering this subject. The question of the use of fæces instead of *B. typhosus* had been discussed again and again, but if there was one substance more variable than another it was fæces, and it was the last thing that one would expect to give satisfactory results in a test of this kind.

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A SIMPLE TEST FOR DIFFERENTIATING BETWEEN COCOA BUTTER AND "GREEN BUTTER."

BY CECIL REVIS AND E. RICHARDS BOLTON.

(*Read at the Meeting*, *April* 2, 1913.)

As at the present time products of varying composition known as "green butters" are frequently used as cocoa butter substitutes, and so may be found in chocolates either alone or, more generally, mixed with genuine cocoa butter, a reliable method of distinguishing these fats, or detecting them in the presence of one another, is necessary, and more particularly so on account of the extreme and usually exact similarity of the analytical figures of these fats to genuine cocoa butter. In addition to the analytical figures, these butters also conduct themselves like cocoa butter towards Björklund's test and other solubility tests so nearly that they do not admit of differentiation.

As the authors feel that this difficulty may have pressed as heavily on some other analysts as it has on themselves, they venture to put forward the following method, which has been of great assistance to them; though, on account of the great varieties of "green butter" which occur, they feel a certain amount of diffidence in regard to its universal application, though believing that in the majority of cases it will give information which cannot be obtained in any other way. It must be clearly understood that this test is not intended to supersede the determination of the ordinary analytical constants, but is to be used in those cases in which the analytical results fail to indicate fats other than cocoa butter.

The test is really a modification of one published by Halphen (*J. Pharm. Chim.*, 1908, **28**, 345). The test as described by him is as follows: One grm. of the absolutely clear filtered fat is dissolved in 2 c.c. of carbon tetrachloride; to 2 c.c. of this mixture is added a solution of bromine in carbon tetrachloride (made by adding bromine to an equal volume of carbon tetrachloride) drop by drop until the colour of the bromine is just permanent; to the mixture are added 3 c.c. of petroleum (sp. gr. 0·700), and the tube stoppered, and allowed to stand twenty-four hours at ordinary room temperature. Halphen states that under these circumstances a solution of cocoa butter remains perfectly clear, whilst "green butters" give a flocculent precipitate, and that in this way 5 per cent. of "green butter" can be detected in cocoa butter.

The authors' experience of this test has not been satisfactory, as several "green butters" gave practically no precipitate at all. It was noticed, however, that on the addition of the bromine to the solution of cocoa butter in carbon tetrachloride, the mixture immediately became turbid, while with "green butters" it remained perfectly clear, and that this turbidity was not soluble in petroleum *ether*, whereas it is in the petroleum suggested by Halphen.

By making a slight modification in the fat solvent, etc., we have found that this method will absolutely differentiate between "green butters" (at least in the case of the many that we have tried) and genuine cocoa butter, and will detect the presence *of 10 per cent. of cocoa butter when mixed with "green butter." The modified test is as follows: One grm. of the clear filtered fat is dissolved in 2 c.c. of a mixture of* equal parts of carbon tetrachloride and petroleum ether (distilling below 40° C.), and 2 c.c. of this solution are placed in a test-tube about 6 inches long and ½ inch in diameter. This tube is cooled in water, and the solution of bromine in carbon tetrachloride (see above) added drop by drop, with constant shaking, until the colour of the bromine is permanent. The greatest care must be taken that only one drop in excess is allowed. The tube is then corked and allowed to stand. If, after the expiration of fifteen minutes, the solution is perfectly clear, cocoa butter is not present, or there is less than 10 per cent. If the solution shows any turbidity, the presence of cocoa butter is indicated, except in the case of one—somewhat rare—cocoa butter substitute obtained from a species of Gutta nut. This one exception, however, does not give quite the same turbidity as cocoa butter, and can easily be distinguished as described below.

The method can be made roughly quantitative by making mixtures of cocoa butter and some solid fat of low iodine value (such as cocoanut oil or cocoanut "stearine" if an actual "green butter" is not to hand), and comparing the turbidities produced by these mixtures and the sample under examination.

After the turbidity has been compared, 2 c.c. of petroleum ether are added to the tubes, which, after mixing by inversion, are allowed to stand all night, when the cocoa-butter turbidity settles out as a very fine canary-coloured precipitate, easily distinguished from the slight flocculent precipitate which "green butters" under these circumstances usually throw down. It is to be also noted that cocoa butter is completely soluble in the carbon tetrachloride-petroleum-ether mixture in the strength given above, whereas "green butters" usually become turbid almost immediately, and on standing for two hours usually throw down a considerable precipitate. Care must therefore be taken that the solution used for the test is quite clear.

The fat mentioned above, which might possibly be mistaken for cocoa butter, may be distinguished from true cocoa butter as follows: The solution of the fat, after treatment with the bromine, is allowed to stand for fifteen minutes, and the turbidity is then carefully examined by transmitted light. The turbidity due to cocoa butter is absolutely non-flocculent, and any appearance of flocculent particles is characteristic of this other fat. If now to the brominated solution are added 2 c.c. of petroleum (fraction of motor spirit distilling between 90° and 100° C.) and the whole mixed, any turbidity due to cocoa butter entirely dissolves, whilst the turbidity due to this other fat remains quite insoluble.

By this means 5 per cent. of this fat may be detected in admixture with 95 per cent. of cocoa butter or "green butter." More than 10 per cent. of this fat produces such a heavy flocculent precipitate that it could not possibly be mistaken.

DISCUSSION.

Dr. DYER asked what were the names of the seeds that yielded "green butter."

Mr. A. E. PARKES, referring to the authors' observation that samples of cocoa butter did not invariably react with Halphen's test, suggested that possibly this difference might be due to variations in the composition of samples from different countries. From the authors' description of the reaction it seemed as though the precipitate were probably a bromine compound of a similar nature to that obtained in the hexabromide test.

The PRESIDENT asked what was the cause of the colour of "green butter."

Mr. BOLTON said that he did not know exactly what the green colour was due to—it was probably chlorophyll—but there were on the market so-called "green butters" which consisted entirely of other fats coloured with an aniline dye. The colour of "green butter" was now, however, frequently taken out in refining, and some of the tasteless products now sold were either colourless or coloured yellow. The origin of the substance was somewhat of a manufacturer's secret, but it was a type of vegetable tallow. The particular type of vegetable tallow that was considered to be the most suitable was now rather scarce, but would probably be more abundant in the near future. The fruit was obtained from the trees only about once in three years.

Mr. REVIS said that the great difficulty in this matter lay in the fact that many cocoa butter substitutes were not cocoanut-oil products. This test, based on that devised by Halphen, was the only one that afforded any assistance, but it must be borne in mind that what it really did was to detect cocoa butter. They hoped that the process would be tried by others, because, although it had proved successful with such samples as they had been able to obtain, that experience might possibly not be universal. As Mr. Parkes had mentioned, cocoa beans and cocoa varied considerably in composition, but in spite of this the composition of cocoa butter was fairly constant. He did not know what the turbidity was due to, but imagined that it must be caused by some action of the bromine on the unsaponifiable matter.

Mr. BOLTON remarked that "green butter" was liable to vary a good deal in composition, because if there were not enough of the kind of fat usually used other fats would be added. He had known shea-nut "stearine" to be sold as "green butter," but that would be readily detected in other ways. He should like again to emphasise the fact that it was only necessary to apply this special test when the usual constants were exactly the same as those of cocoa butter.

The PRESIDENT asked whether "green butter" was as wholesome as cocoa butter.

Mr. BOLTON said that it seemed to be perfectly wholesome and harmless, and there was no reason whatever to suppose that it contained anything deleterious. With regard to the question of its comparison with cocoa butter, it must be remembered that "green butter" was subjected to all the processes of the modern refiner and was usually tasteless, whereas cocoa butter was simply pressed out of the beans, filtered or not, as the case might be, and clarified.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Use of Miscibility Curves in the Examination of Spirits of Camphor. H. Rosset. (*Ann. Chim. anal.*, 1913, **18**, 49-56.)—The author has extended the methods described previously by Louise (ANALYST, 1907, **32**, 365; 1910, **35**, 322; 1911, **36**, 556) to the analysis of alcoholic solutions of camphor, and shows that it is possible to estimate both the camphor and the alcohol from the miscibility curves obtained. The alcohol is found from curves plotted from the results given by mixtures of alcohol and standard petroleum spirit, and the amount of camphor from the miscibility curves of mixtures of alcohol and nitrobenzene. W. P. S.

Polenske "Difference Value" for the Detection of Certain Animal Fats and its Theoretical Basis. A. Bömer and R. Limprich. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1913, **25**, 367-386.)—The method described by Polenske (ANALYST, 1907, **32**, 382; 1908, **33**, 476) is of use for the detection of beef or mutton fat in lard when the quantity of the former is not less than from 15 to 20 per cent., and the method depends on the difference in composition of the glycerides of lard and beef fat. The value is chiefly affected by the palmitodistearin present in the fats, the α-palmitodistearin of lard giving the fat a "difference value" of 18·4, whilst β-palmitodistearin is the source of the "difference value" of 11·8 in the case of beef or mutton fat. No advantage is derived from determining the "difference value" of mixed glycerides which crystallise from an ethereal solution of the fats.

W. P. S.

Mixed Glycerides of Palmitic and Stearic Acids Present in Lard. A. Bömer. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1913, **25**, 321-353.)—By repeated fractional precipitation and crystallisation of lard from ether, the author has isolated the saturated glycerides of this fat. Tristearin is not present in lard, and in this respect the latter differs from beef and mutton tallows. The insoluble glyceride obtained from lard is palmitodistearin and not heptadecyldistearin, as stated by Kreis and Hafner (ANALYST, 1904, **29**, 259). Heptadecyldistearin could not be detected in lard. As the palmitodistearin obtained from lard differs in its melting-point and crystalline form from the palmitodistearin derived from mutton tallow, it is probable that the former is α-palmitodistearin and the latter β-palmitodistearin. Lard was also found to contain a second saturated glyceride—namely, stearodipalmitin. The lard used in the experiments yielded about 3 per cent. of α-palmitodistearin, melting-point, 68·5° C., and about 2 per cent. of stearodipalmitin, melting-point 58·2° C. W. P. S.

Lecithin Preparations and the Estimation of Lecithin. R. Cohn. (*Zeitsch. öffentl. Chem.*, 1913, **19**, 54-62; through *Chem. Zentralbl.*, 1913, I., 1129-1130.)—The estimation of lecithin may be divided into three parts—namely, the extraction of the lecithin, its purification, and the estimation of the phosphorus in the product. For the extraction of the lecithin from 1 to 2 grms. of commercial preparations of lecithin, or from 5 to 20 grms. of a food material containing lecithin, are extracted for several hours with two successive quantities of 100 c.c. of 96 per cent. alcohol, the first extraction being carried out at the ordinary temperature and the second at the boiling temperature of the alcohol, a reflux apparatus being used in this case. The residue is then ground up with sand, extracted once more with alcohol, and then boiled for two hours with about 100 c.c. of chloroform. When dealing with fatty substances, the chloroform extraction may be made with advantage immediately after the first extraction with cold alcohol. The ease with which lecithin may be extracted from a preparation depends largely on the treatment to which it has been subjected during manufacture; prolonged heating or lengthy storage renders the lecithin less soluble, and in certain cases, the extraction with hot alcohol must be continued for, say, twenty hours, before phosphorus compounds cease to be extracted. After the alcohol and chloroform extracts have been evaporated, the residue obtained is boiled for two hours with 100 c.c. of chloroform in order to separate the lecithin from glyceryl-phosphoric acid and free phosphoric acid; the chloroform solution is then filtered and evaporated. The quantity of phosphorus in this residue is then estimated by oxidising it with nitric acid and sulphuric acid, or igniting it with the addition of magnesium oxide, or a mixture of sodium carbonate and potassium nitrate, precipitating the resulting phosphoric acid with molybdic acid solution, and converting the molybdate precipitate into ammonium magnesium phosphate in the usual way.

Commercial preparations of lecithin frequently contain less than the guaranteed quantity of lecithin; the author considers that a substance sold as pure lecithin should contain, at the least, from 90 to 95 per cent. of lecithin, and that the quantity of the latter in any preparation should be within 10 per cent. of the amount claimed by the manufacturers to be present. The quantity should be expressed as lecithin itself, and not as lecithin-albumin or lecithin-protein, as these are of variable composition, and may contain from 5 to 30 per cent. of lecithin.

The author does not attempt to solve the question whether yolk of egg contains lecithin in chemical combination, but points out that, as lecithin may be almost completely extracted from yolk of egg by means of cold alcohol, the presence of combined lecithin is improbable. The insolubility of lecithin under certain conditions is possibly due to adsorption by proteins. W. P. S.

Effect of Boiling on the Physico-Chemical Behaviour of Human Milk, Cow's Milk, and Buttermilk. P. Grosser. (*Biochem. Zeitsch.*, 1913, **48**, 427-432.) —The experiments were undertaken to determine whether the two kinds of milk are affected differently by boiling, and if it is possible to find a difference between raw and boiled milk by physico-chemical methods. The milk, from which the cream was removed by means of a separator, was filtered through a Bechold ultrafilter by means of compressed nitrogen at 6 atmospheres. The filtrate, which was free from colloids, was examined with regard to depression of freezing-point, and nitrogen, phosphorus, and lime content. The results indicate that boiling does not affect the freezing-point, and in the case of cow's milk scarcely affects the phosphoric acid and nitrogen. In human milk the phosphorus and nitrogen sink considerably on boiling. The lime content of the ultrafiltrate of both milks diminished on continued boiling, the diminution being greater in human milk. In the case of butter milk, boiling produced no change in the values determined. E. W.

Detection of Saffron in Confectionery. C. Martini. (*Staz. sperim. agrar. ital.*, 1912, **46**, 18-24; through *Chem. Zentralbl.*, 1913, I., 1068.)—The finely powdered confectionery is extracted for twenty-four hours in the cold with 70 per cent. alcohol with frequent agitation. Fifty grms. of the powdered residue are then boiled for fifteen minutes with 100 c.c. of 70 per cent. alcohol under a reflux condenser on the water-bath. After filtration, the residue is again boiled with fresh alcohol, the united extracts are concentrated on the water-bath; it is then exhausted with ether. The extract, after evaporating the ether, is boiled with 98 per cent. alcohol, and the latter solution evaporated. The product is then tested by the well-known colour reaction for saffron, with sulphuric and nitric acids. J. F. B.

Chemical Composition of Authentic Vanilla Extracts, together with Analytical Methods and Limits of Constants. A. L. Winton and E. H. Berry. (*U.S. Dept. Agric.*, *Bureau of Chem.*, Bull. No. 152.)—After a discussion of the commercial origin of various kinds of vanilla beans, and the processes of manufacture of vanilla extract, methods of analysis are fully described, including the determination of vanillin and coumarin by the modified Hess and Prescott method (*J. Amer. Chem. Soc.*, 1899, **21**, 256; *ibid.*, 1902, **24**, 1128; *ibid.*, 1905, **27**, 719; *U.S. Dept. Agric.*, *Bureau of Chem.*, Bull. 132, p. 109, and Bull. 137, p. 120); the normal lead number (Winton and Lott method, *U.S. Dept. Agric.*, *Bureau of Chem.*, Bull. 132, p. 110, and Bull. 137, p. 120); the colour value of the extract; the residual colour after precipitation with lead acetate; and the colour insoluble in amyl alcohol (Tolman and Hillyer, *Bull.* 122, p. 206; Bull. 132, p. 90). A large number of samples of known origin were examined in the laboratory by preparing extracts from them, following the method employed by the U.S. Pharmacopœia, in order to learn the influence of variety, grade, and length of bean on the composition of the vanilla extract. In discussing the results of these analyses (all the figures of which are recorded) it is observed that the extracts from Ceylon beans were so abnormal and variable in composition as to indicate either unusual curing or previous extraction.

Vanillin.—The range in vanillin content ran from 0·11 to 0·31 grm. per 100 c.c., and is somewhat greater than has usually been thought possible; but the figure 0·25 stated by Leach to be regarded with suspicion refers to extracts on the commercial scale and not in the laboratory. The minimum figure (0·11) was found in Tahiti extracts prepared from undried beans.

Normal Lead Number.—The variation in this value (0·4 to 0·74) is less than that of any other constant. The constituents yielding the precipitate appear to be more easily soluble than the vanillin and the colour. This determination not only serves to distinguish a true extract from a solution of vanillin, but is also of value taken along with a coumarin estimation as a means of detecting the presence of Tonka extract.

Colour Values of Extract and Lead Filtrate.—The colour is the most variable and difficultly extractable constituent. As a means of detecting caramel, the percentage of colour left in the lead filtrate is most significant. The maximum found in these samples (excluding Ceylon) was 8 per cent. red and 10 per cent. yellow, these figures being far surpassed by caramel-coloured extracts. As regards length of bean, the differences found, although not marked, point to a slight inferiority of the shorter beans.

The next section deals with vanilla extracts prepared with different solvents and solvent mixtures in the laboratory, the standard solvent being 60 per cent. alcohol and sugar, the other solvents examined being (1) 60 per cent. alcohol alone; (2) 60 per cent. alcohol and glycerol; (3) 35 per cent. alcohol alone; (4) 35 per cent. alcohol and sugar; (5) 35 per cent. alcohol and glycerol. The 35 per cent. extracts were highly unsatisfactory, owing to the extraction of gelatinous material which clogged the percolators. With the same strength of alcohol the only noteworthy difference was found to be that the glycerol extracts were more strongly coloured, thus explaining the popularity of glycerol for commercial extracts.

Standard Vanilla Extract.—The following tentative limits of composition for standard vanilla extract (10 grms. of beans to 100 c.c.) appear to be warranted by the results obtained:

Vanillin, 0·10 to 0·35 grm. per 100 c.c.
Normal lead number, 0·40 to 0·80.
Per cent. of total colour in lead filtrate, not more than 10 per cent. red, or 12 per cent. yellow.
Ratio of red to yellow in the extract, not less than 1 : 2·2.
Colour insoluble in amyl alcohol, not more than 40 per cent.
(*Cf.* O. Folin and W. Denis, ANALYST, 1912, **37**, 501.) H. F. E. H.

Direct Estimation of Water in Foods, etc., by Distillation. F. Michel. (*Chem. Zeit.*, 1913, **37**, 353-355.)—This method has been described previously by Aschmann and Arend (ANALYST, 1907, **32**, 21) who have employed it for the estimation of water in butter and other fats, and the author now shows that it may be applied to almost all classes of foods. He recommends the use of a mixture consisting of 1 volume of toluene and 2 volumes of xylene, about 150 c.c. of this mixture being employed for 20 grms. of the substance under examination. Ordinary paraffin, having a boiling-point above 100° C., may also be used. The distillate is collected in a narrow graduated cylinder, and, at the end of the distillation, any drops of water remaining in the condenser may be transferred to the receiver by means of a rubber-tipped rod or feather dipped in xylene. In order that the volume of the water in the receiver may be determined accurately, the contents of the receiver may be submitted to centrifugal action. A correction must be made on the volume of water observed owing to the change of meniscus caused by the hydrocarbon layer above the water layer; for instance, if a quantity of xylene be poured on the surface of 5 c.c. of water contained in a narrow cylinder, the water will then show an apparent volume of 4·85 c.c. Consequently, 0·15 c.c. is added to the volume of water found. The author finds that a further quantity of 0·03 c.c. must be added for each c.c. of water collected in the receiver in order to allow for loss during the distillation. When these allowances are made, the error of the method is not greater than ±0·2 per cent. W. P. S.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Analysis of Human Bile. E. v. Czyhlarz, A. Fuchs, and O. v. Fürth. (*Biochem. Zeitsch.*, 1913, **49**, 120-130.)—About 30 c.c. bile are required. This is divided into three portions: for the determination of moisture, the bile pigment, and the bile acids—mucin, fatty matter, and cholesterol.

The moisture is determined on 3 c.c. by drying at 100° C. until constant weight. For the determination of bile pigment, 5 c.c. are heated with 5 c.c. 10 per cent. sodium hydroxide solution on the water-bath for half an hour, whereby the bilirubin is converted into biliverdin. Thirty c.c. of 95 per cent. alcohol are then added to the hot liquid, the precipitate filtered off, and to the filtrate dilute hydrochloric acid is added drop by drop until there is a slight acidity. The clear and intensely emerald green-coloured liquid thus obtained is compared colorimetrically with a 0·02 per cent. alcoholic biliverdin solution in a Dubosq colorimeter. The third portion (about 22 c.c.) is extracted three times with ether. A small quantity of alcohol may be added to suppress emulsification, which may be very troublesome, and it is sometimes necessary to wait several hours to obtain a clean separation. The extract is freed from ether, the residue evaporated on the water-bath with 20 c.c. of freshly prepared sodium ethylate solution, dissolved in water, and extracted with ether to separate cholesterol from the soaps. The ethereal layer is then evaporated, and the residue dissolved in 10 c.c. pure chloroform. Should there be any residue, it is taken up with a little water and added to the soap fraction. The cholesterol in the chloroform solution is determined colorimetrically by the method of E. Schulze (*Zeitsch. physiol. Chem.*, 1890, **14**, 503), and Grigant (*Compt. rend. Soc. Biol.*, 1910, **68**, 827), for which purpose 10 c.c. of the chloroform solution are treated with 2 c.c. commercial acetic anhydride (reaction not given by pure substance), and 10 drops concentrated sulphuric acid, shaken and allowed to stand a few minutes. This liquid is then compared colorimetrically with a 0·05 per cent. cholesterol solution treated in a similar manner.

For the estimation of the higher fatty acids, the aqueous soap solution (see above) is acidified with sulphuric acid, extracted with ether, the ether extract evaporated, and the residue dried at 100° C. and weighed.

Mucin is estimated in the aqueous portion of the main fraction. This is treated with 80 c.c. 95 per cent. alcohol, and the precipitated mucin collected on a hardened filter, and washed with alcohol. If the mucin precipitate is large, it can be removed from the filter while still moist, dried at 100° C., and weighed; otherwise the nitrogen is estimated by Kjeldahl's method, the nitrogen content of mucin being taken as 16·14 per cent. The alcoholic filtrate is evaporated on the water-bath; the residue, taken up in 20 c.c. water, is transferred to a large pressure bottle, decomposed with two and a half times its weight of 60 per cent. caustic potash, and the closed bottle heated for forty-eight hours in a boiling water-bath. After cooling, the contents of the bottle are transferred to a beaker, diluted with twice its volume of water, and made strongly acid with concentrated hydrochloric acid, and allowed to stand for one day. The cholic acid precipitate is then collected, washed well with water, and then dissolved from the filter by means of hot 95 per cent. alcohol. The alcoholic solution is evaporated to dryness, and the residue dissolved in a small quantity absolute alcohol, and filtered to remove any inorganic salts, and the filtrate evaporated to dryness, dried at 100° C. and weighed.

The author gives numerous results obtained by the above methods with abnormal biles. E. W.

Study of the Oxydation Products of Cholesterol. I. Lifschütz.

(*Biochem. Zeitsch.*, 1913, **48**, 373-410.)—The author finds that in various diseases the oxycholesterol content of the blood differs from that of the normal tissue. Thus the blood-fat of a man in a comatose condition contained no trace of oxycholesterol, whilst in cases of diabetes and syphilis the content was below the normal; and of several blood sera of insane, oxycholesterol was absent in two and present only in small quantity in the remainder. The oxycholesterol is detected by the coloration produced on adding sulphuric acid to an acetic acid solution, and is determined quantitatively by comparing this colour spectrometrically with that produced in a standard solution of known oxycholesterol content. The standard solution is prepared by diluting 1 c.c. of a 0·1 per cent. solution of oxycholesterol in chloroform with 2 c.c. glacial acetic acid. The material to be examined is similarly dissolved, the amount employed varying with its oxycholesterol content; for unsaponifiable matter the author recommends a 0·4 to 0·6 per cent. chloroform solution. To each solution 7 to 8 drops of strong sulphuric acid are added, the liquids mixed, and after standing ten to fifteen minutes, 1 to 2 drops of a 5 per cent. solution of ferric chloride in glacial acetic acid are added. The two liquids are then examined by the spectroscope, and the more concentrated solution diluted until the two liquids yield spectra of equal intensity. The oxycholesterol can be prepared by treating a solution of 2 grms. cholesterol in 100 c.c. glacial acetic acid with 2 grms. benzoyl peroxide, and boiling until the light yellow liquid assumes a slight brownish tint. The cooled liquid is poured into water, the reaction product separated, repeatedly boiled with water, and then boiled with 50 c.c. $\frac{N}{2}$ potassium hydroxide for forty-five minutes. The liquid is then diluted with water and extracted with ether. The oxycholesterol thus obtained is stable if protected from light and moisture, and is a suitable standard for spectrometric purposes. E. W.

Determination of Pepsin Activity. S. V. Bogdóndy. (*Zeitsch. physiol. Chem.*, 1913, **84**, 18-29.)—Sixty c.c. of a casein solution prepared by mixing 17·5 grms. casein with 250 to 300 c.c. water, adding 27·5 c.c. $\frac{N}{1}$ hydrochloric acid, diluting to 500 c.c. and heating to 40° C. to obtain perfect solution, are mixed with the pepsin, heated to 40° C. for a known time, and the unchanged casein precipitated by adding 30 c.c. of a reagent containing 150 grms. sodium sulphate, 50 grms. magnesium sulphate, and 100 c.c. 96 per cent. alcohol per litre. The mixture is made up to 100 c.c. with water, filtered, and the rotatory power of the filtrate measured. This rotatory power, though admitted to be not exactly proportional to, is yet a measure of, the activity of the pepsin, and the results obtained agree well with those given by known methods. E. W.

Application of Precipitin Reactions to Seed Identification. Zade. (*Bull. Agric. Intell. and of Plant Diseases*, 1913, **4**, 200-201.)—It is not possible to distinguish, without previous cultivation experiments, a winter from a summer wheat, nor an awned from an awnless variety; the nature of the seeds of leguminous fodder crops also is only ascertained, and then not with absolute certainty, from the impurities found among them. Relander has experimented with the object of adopting the precipitin method for seed identification. The manner of proceeding is as follows: A sample of wheat, for instance, is reduced to a fine powder and treated with a given volume of physiological salt solution. The extract is filtered and injected in small quantities, repeated at fixed intervals (three to ten days) into the blood of the animal which is the subject of the experiment. A small amount of the blood of the animal is then taken, the immunised serum separated by centrifuging, and filtered perfectly clear. On adding to the filtrate some drops of the original wheat extract, a precipitate is obtained which is not produced if extracts are injected which have been prepared from other seeds. Relander by this method found that the serum of a rabbit which had been injected with an extract of two-rowed barley reacted in the presence of a similar extract (the reaction being more energetic for the same variety than for different ones), while there was no reaction in the case of six-rowed barley. The same method rendered it possible to distinguish Italian and American clover-seed from Finnish and Norwegian, but the two latter are indistinguishable from one another. H. F. E. H.

Biological Considerations in Relation to Seed-Testing. D. Schaffnit. (*Bull. Agric. Intell. and of Plant Diseases*, 1912, **3**, 2403.)—The usual method of testing seeds determines only the germinating power when placed under favourable conditions. Many seeds which will germinate are deficient in "shooting power" (Triebkraft)—that is to say, they fail to emerge above the surface of the soil when grown at a depth of from 3 to 8 cm. Some seeds showing 100 per cent. germinating capacity often yielded 60 per cent. or less of shooting power, thus explaining the bad result after sowing in the field of seeds which by laboratory tests have been found faultless. Such weakness is a special characteristic of small seeds which have at their disposal a less quantity of vital energy and a proportionately smaller quantity of reserved substances. Failure to emerge from the surface of the soil after germination may be due to the attacks of *fusarium* and other micro-organisms. Chemical substances such as sulphate of copper or formaldehyde, or excessive heating in the destruction of smut (*ustilago*), will also bring about a similar result. To obtain uniform results in the testing of shooting power, the following constant conditions of cultivation are necessary: (1) Regularity and uniformity of the medium in which sowing is effected; (2) determined depth of sowing; (3) constant quantity of water in the medium; (4) constant temperature. The best medium was found to be coarse brick-powder freed from the fine dust by sifting. Each time before use it is heated for two hours to 150° C., then cooled, and mixed with 20 per cent. of water. Cereal seeds should be sown at a depth of 3 cm. The germinating boxes are covered with a glass bell jar, thus insuring a constant moisture content. The temperature should be kept at 15° C. H. F. E. H.

ORGANIC ANALYSIS.

Determination of Chlorides and Bromides in Organic Liquids. S. v. Bogdóndy. (*Zeitsch. physiol. Chem.*, 1913, **84**, 11-15.)—The substance is decomposed by heating with a mixture of concentrated and fuming sulphuric acid, as used in Kjeldahl's determinations, to which 10 grms. copper sulphate and 80 grms. potassium sulphate are added per 250 c.c. The decomposition is carried out in a flask connected with two suitable absorption vessels containing silver nitrate dissolved in 20 per cent. nitric acid. A current of air is drawn through the apparatus, and the heating continued until the residue becomes transparent and blue. The silver precipitates are then treated in the usual manner. E. W.

Accuracy and Limitations of Coal Analysis. A. C. Fieldner. (*Chem. Engineer*, 1913, **17**, 50-65.)—A paper communicated by permission of the Director of the United States Bureau of Mines, and supported by statistics from the laboratory records of that department. It is stated that duplicate determinations of "moisture" seldom vary by more than 0·15 per cent., when the ¼-inch mesh sample is air dried at 30° to 40° C., and the air-dried sample reduced to 60 mesh in a ball mill, and 1 grm. dried for one hour at 105° C. Duplicate determinations of "ash" will agree within 0·2 per cent. if a standard temperature be adopted, but the difference between a low and a bright red heat may lead to differences of more than 1 per cent. Determinations of "volatile matter" made by the same analyst in the same laboratory may differ by 0·5 per cent., but between the results of different laboratories the difference may rise to nearly 3 per cent., and these wide differences most often occur on samples of anthracite, in which the amount of volatile matter is so small that the two numbers returned may approximate the ratio of 2 : 1. With soft coals such wide differences are less common and are less serious as the figure to be returned is of the order of 30 per cent., but for anthracites the adoption of a standard temperature is essential if the figures are to possess any value at all. A temperature of 950° to 1,000° C. is recommended for all coals.

Carbon can be determined with an error not exceeding 0·3 per cent., hydrogen with an error of less than 0·05 per cent., and nitrogen within 0·03 per cent., but the value of the results of ultimate analysis as a means of calculating the calorific value is reduced by the uncertainty attaching to the figure for oxygen. This figure has to bear the sum of all the errors incurred in the determination of carbon, hydrogen, nitrogen, sulphur, and ash. Of these errors, by far the most serious is that due to the difference between the "ash" as determined, and the mineral matter as it occurs in the coal. In spite of this it is shown that the application of Dulong's formula to true coals rarely leads to values which differ by more than 1·5 per cent. from the values determined by the bomb calorimeter. With lignites and peat, Dulong's formula leads to results much below the truth. The limit of accuracy of carefully conducted work with a bomb calorimeter is not put above 0·5 per cent., but it is stated that this is less than the usual error of sampling, which, without special precautions, may be 1 per cent. In the case of coal from one mine and seam, a fairly accurate calculation of the calorific value can be made from the percentage of ash and moisture in the sample under consideration, and the calorific value of the "coal substance" of that seam of coal as determined previously. The error of such a calculation rarely amounts to 1 per cent.

For the fine grinding of coal samples, the use of a porcelain ball mill is strongly recommended. The abrasion of the pebbles is negligible in its influence on the percentage of ash (0·004 per cent.), but the porcelain cylinders suffer abrasion to an extent which makes it advisable to grind as much as 500 grms. of the sample, so as to reduce the percentage error to 0·04 per cent. or less, which is negligible. These figures are supported by results representing the grinding of nearly half a ton of coal (more than 1,000 samples). The use of a tool steel diamond mortar may lead to the over-estimation of the ash of a hard foundry coke by more than 1 per cent., whilst the use of a chilled cast-iron bucking-board may lead to errors in excess of 4 per cent.

G. C. J.

Chemical Examination of Liquid Fuels. W. H. Paterson. (*J. Soc. Chem. Ind.*, 1913, **32**, 218.)—An "oil fuel," or oil for internal combustion engines, must be volatile, and must not cause corrosion or other difficulties in the cylinder. For a "fuel oil," or oil to be used in firing boilers, cheapness, comparatively high flash-point, and high calorific value are more important. In addition to petrol, alcohol and benzene, or mixtures of them, may be used in certain internal combustion engines; but in competition with coal only crude petroleum, or petroleum residues, tar oils, products from animal or vegetable sources, and oils from lignite, peat, wood, or shale, can be considered. A list of eighteen liquid fuels, none exceeding £3 per ton in price, with their sp. gr., carbon hydrogen, sulphur, oxygen and nitrogen, gross, net, and calculated calorific values, open flash-point and burning-point, and a table of fractions obtained at various temperatures, are given. Some of these oils have been successfully used in the Diesel engine, and most of them are probably suitable. As fuel oils, their efficiency depends on their calorific value. The importance of the corrections for the latent heat of steam and for acid in obtaining the net from the gross calorific value is emphasised. The viscosity is an important factor, especially in oils for the Diesel engines, as too mobile an oil leaks through the valves. At low temperatures the fluidity of some oils increases rapidly with the temperature. For petroleum products there is a general correlation between low sp. gr., low flash-point, high hydrogen content, and low boiling-point. O. E. M.

New German Regulations for the Official Examination of Inks. F. W. Hinrichsen. (*Chem. Zeit.*, 1913, **37**, 265-267.)—According to the official German regulations of May, 1912, "documentary ink" is an iron gall ink containing at least 27 grms. of anhydrous gallotannic and gallic acids, and not less than 4 grms. or more than 6 grms. of iron per litre (ratio 4·5 : 1 and 6·75 : 1). It must keep at least fourteen days in the inkpot, without deposit, etc., must flow readily from the pen, and produce writing which, after eight days' exposure to light and air, remains deep black when washed with water, 85 per cent. alcohol, and 50 per cent. alcohol. An ordinary iron-gall writing-ink must answer the same requirements, but may contain less tannin (18 grms.), with not less than 2·6 grms. or more than 4 grms. of iron per litre (ratio as before). Gallotannic and gallic acids are estimated by shaking the ink with ethyl acetate, evaporating the extract, and weighing the residue. The latter is regarded as consisting of gallotannic and gallic acids when 0·1 grm. thereof absorbs at least 0·5 grm. of iodine in the presence of 2 grms. of sodium bicarbonate. The method of Rothe and Hinrichsen is suitable for the estimation: Ten c.c. of the ink are treated with 10 c.c. of concentrated hydrochloric acid, and shaken with successive portions of 50 c.c. of ethyl acetate, preferably in a shaking apparatus, until the aqueous layer when neutralised with sodium carbonate gives no reaction for gallotannic or gallic acids with ferrous sulphate and ferric chloride. The united extracts are shaken several times with fresh portions of 10 c.c. of a semi-saturated solution of potassium chloride, to remove any dissolved iron salts, and are then evaporated *in vacuo* and the residue transferred by means of water to a tared crucible, and dried at 105° to 110° C., or, better, *in vacuo* at 60° C., until constant in weight. In estimating the iodine absorption, 0·1 grm. of the residue is mixed in a stoppered flask with 2 grms. of sodium bicarbonate and a measured quantity (25 to 50 c.c.) of standard iodine solution (about 50 grms. per litre). Simultaneously a blank estimation is made, and the next day the contents of both flasks are titrated with sodium thiosulphate solution. Iron is estimated by igniting the residue from 10 c.c. of the ink until free from carbon, and then heating it with 1 to 2 c.c. of concentrated hydrochloric acid (sp. gr. 1·124) in a covered crucible on the sand-bath until dissolved. From 1 to 2 c.c. of chlorine-water are next added, the solution evaporated to dryness, the residue dissolved in about 0·5 c.c. of warm strong hydrochloric acid, and the solution diluted with about 20 c.c. of water. About 1 grm. of potassium iodide is then added, and the separated iodine immediately titrated with $\frac{N}{10}$ thiosulphate solution, whilst the liquid is meanwhile heated to 55° C., to promote the further separation of iodine. In testing the permanency of the writing, the ink is made to flow over standard paper fixed in a frame inclined at an angle of 45°, the pipette being placed in a rest so that it is held at a definite angle towards the paper. Similar colour bands are made upon the same paper with Schlüttig and Neumann's standard ink. This contains 23·4 grms. of tannin, 7·7 grms. of crystalline gallic acid, 30 grms. of ferrous sulphate, 10 grms. of gum arabic, 2·5 grms. of hydrochloric acid, and 1·0 grm. of carbolic acid, per litre. It is allowed to stand for four days at 10° to 15° C., and then decanted from the slight sediment. For comparison with any given ink, it is coloured with a suitable provisional dyestuff to match the sample. The papers with the coloured bands of standard ink and the ink under examination are exposed to the air for eight days in diffused light, and are then cut horizontally into strips. One of these is immersed in water, a second in 85 per cent. alcohol, and a third in 50 per cent. alcohol. None of the bands of ink should appear perceptibly fainter after being treated in this way for several days. C. A. M.

Red Cabbage Extract as Indicator for Measuring Hydrogen Ion Concentration. L. E. Walbum (*Biochem. Zeitsch.*, 1913, **48**, 291-296.)—The indicator is prepared by forty-eight hours' extraction of 500 grms. finely-cut red cabbage with 500 grms. 96 per cent. alcohol, and subsequent filtration through paper. The author measured the hydrogen ion concentration of a number of protein solutions by means of this indicator, and compared the results with those obtained electrometrically and those obtained by a number of indicators of the azo group, and found that the colorimetric estimation by means of red cabbage extract gives values agreeing satisfactorily with those found electrometrically. The indicator can be used for hydrogen ion concentrations ranging from $\mathrm{PH}^{\bullet}$ = about 2·0 to $\mathrm{PH}^{\bullet}$ = about 4·5.

The indicator gives satisfactory results even in presence of considerable quantities of proteins. Neutral salts (up to 0·5 molecule sodium chloride), toluene, and chloroform, exercise no disturbing action. E. W.

Nomenclature of Mixed Glycerides and the Synthetic Preparation of α-Distearin and β-Palmitodistearin. A. Bömer and R. Limprich. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1913, **25**, 354-366.)—It is suggested that, in the case of mixed glycerides where different fatty acid radicles are combined with the glyceryl radicle, the position of the acid radicle should be denoted. For instance, the two isomeric glycerides containing one palmitic and two stearic radicles may be described as follows:

α-position : $CH_2.O.C_{16}H_{31}O$	α-position : $CH_2.O.C_{18}H_{35}O$
β-position : $CH.O.C_{18}H_{35}O$	β-position : $CH.O.C_{16}H_{31}O$
γ-position : $CH_2.O.C_{18}H_{35}O$	γ-position : $CH_2.O.C_{18}H_{35}O$
α-palmito-βγ-distearin.	β-palmito-αγ-distearin.

As the α and γ positions are of the same value, it is sufficient to term these two glycerides α-palmitodistearin and β-palmitodistearin, respectively. When three different fatty acid radicles are present in the same molecule, their position may be denoted in the same manner.

The authors have prepared α-distearin from α-dichlorhydrin and potassium stearate, using Guth's method (*cf.* ANALYST, 1903, **28**, 152, 359), and found that a considerable quantity of tristearin was produced at the same time; the α-distearin had a melting-point of 78·5° C. Tristearin, and possibly stearodipalmitin, was also formed when β-palmitodistearin was prepared synthetically from α-distearin and palmitic acid. The synthetic β-palmitodistearin melted at about 63° C., and corresponded in other respects with the palmitodistearin isolated from mutton tallow. W. P. S.

Effect of Certain Pigments on Linseed Oil. Manganese Content of Raw Linseed Oil. E. W. Boughton.

(*U.S. Dept. Agric.*, *Bureau of Chem.*, Circular No. 111, February 15, 1913.)—In order to determine the effect of pigments, without driers, upon raw linseed oil, mixtures were made and exposed to diffused light in stoppered jars for periods of one and two years, samples of the mixtures being examined at the end of each period. The oil used contained 0·13 per cent. of ash, and had a sp. gr. of 0·934 at 15·6° C.; acid value, 1·7, and iodine value, 179·6. It was mixed with the following pigments: White lead, kaolin, Indian red, flaked graphite, magnetic oxide (black), zinc yellow (chromate), artificial graphite, zinc white, chrome yellow, chromium oxide (green), and lampblack, the proportions ranging from 16 per cent. (lampblack) to 72 per cent. (white lead). The oils separated from the paints showed slightly reduced iodine values in most cases, the greatest reductions being observed with the so-called "inert" pigments, kaolin and Indian red. In no instance, however, did the iodine value fall below that sometimes given by genuine samples of linseed oil, the minimum value obtained being 171·6 with the oil from the kaolin mixture after two years. The ash yielded by the separated oils did not indicate that any appreciable amount of pigment had been dissolved, except in the case of the white lead paint, the oil of which yielded 0·35 per cent. of ash after one year, and 0·40 per cent. after two years. The sp. gr. of most of the oils showed a decided increase, the highest values, 0·939 to 0·940, being obtained with oils from the mixtures containing white lead, Indian red, kaolin, and artificial graphite. The values obtained after two years, however, were but little lower than those given after one year, and from this it would seem that the air in the jars had but little influence. In many of the samples the sp. gr. exceeded the upper limit for raw linseed oil of 0·936 at 15·6° C. (*Proc. Amer. Soc. Test. Mat.*, 1909, **9**, 164), and this indicates that if the oil separated from a mixed paint after storage shows a sp. gr. approximating the lower limit for pure linseed oil (0·932) it may suggest the presence of other oils of lower specific gravity. Some bleaching of the linseed oil had been effected by white lead, Indian red, chrome yellow, and chromium oxide, and particularly by kaolin, but the other pigments had caused no appreciable change in the colour of the oil. Similar analytical results were obtained in the examination of oil from mixtures containing sufficient white lead to form a paste, but here also no driers or thinning agents had been used. It was found that white lead apparently entered more closely into combination with the oil than lead chromate.

Manganese in Raw Linseed Oil.—The proportion of ash and manganese in sixteen samples of oil of undoubted purity was estimated, the latter by the bismuthate method. The ash ranged from 0·02 to 0·21 per cent., and the manganese from a faint trace to 0·0008 per cent. of the oil. C. A. M.

Permanganate [Alkaline] in the Estimation of Some Organic Compounds. C. M. Pence.

(*J. Ind. and Eng. Chem.*, 1913, **5**, 218-220.)—In alkaline solution, phenol, pyrocatechol, resorcinol, quinol, salicylic acid (but not benzoic acid), and salol are completely oxidised by permanganate to carbon dioxide and water. The following method is recommended for the estimation of any one of them in absence of any other substance which reduces permanganate : A suitable quantity of the substance, brought into solution if necessary by means of $\frac{N}{2}$ sodium hydroxide, is added to 50 c.c. of $\frac{N}{10}$ permanganate containing 3 or 4 grms. of sodium bicarbonate in solution. The mixture is boiled for five to ten minutes, cooled to about 60° C., acidified with dilute sulphuric acid, allowed to stand about two minutes, and then cooled to room temperature. To the cool and diluted solution about 1 grm. of potassium iodide is added, and the excess of permanganate taken is calculated from the amount of $\frac{N}{10}$ thiosulphate required to reduce the liberated iodine. The extreme error of the method is ±0·5 per cent. on the amount of phenol or other reducing substance present. G. C. J.

Direct Estimation of Oxygen in Organic Compounds. M. C. Boswell. (*J. Amer. Chem. Soc.*, 1913, **35**, 284-290.)—The method is based upon the fact that when an organic substance is heated in a quartz tube in a current of pure hydrogen, and the products of the reaction are passed over charcoal at a white heat, the whole of the oxygen in the original compound is converted into water, carbon dioxide, and carbon monoxide. The water is absorbed in tubes containing pumice moistened with sulphuric acid, the carbon dioxide in soda-lime, whilst the carbon monoxide is estimated by passing the residual gas through iodine pentoxide, and absorbing in soda-lime the carbon dioxide thus produced (ANALYST, 1912, **37**, 106). The quartz tube for the combustion is about 980 mm. long by 13 mm. internal diameter, and it is lightly packed with fragments of charcoal for a length of 400 mm. from a point 135 mm. from one end. This tube is heated in an ordinary combustion furnace from which the iron trough and tiling have been removed, and, further to prevent transference of heat to the substance during the preliminary heating of the charcoal, two guards of asbestos board are fitted across the furnace, one 15 mm. from the end of the charcoal layer, and the other 30 mm. from the first. The hydrogen is generated electrolytically, and oxygen is removed by passing the gas through a quartz tube (360 mm. × 13 mm.) containing a tightly wound coil of copper gauze which is heated to white heat. After leaving this, the hydrogen is dried by passing through calcium chloride, then through sulphuric acid, and finally through phosphorus pentoxide, after which it enters the combustion tube. The tared and other absorption-vessels are arranged in the following order : (1) A U-tube with sulphuric acid on pumice ; (2) soda-lime tube ; (3) U-tube with sulphuric acid on pumice to retain moisture carried over from (2) ; (4) modification of Levy's iodine pentoxide apparatus, which during the estimation is heated in an oil-bath at 160° to 170° C. ; (5) U-tube with sulphuric acid on pumice to absorb any moisture liberated when any iodic acid is reduced by the carbon monoxide ; (6) soda-lime tube ; (7) U-tube with sulphuric acid on pumice ; and (8) straight guard tube of calcium chloride. About 0·3 grm. of the substance is weighed into a silica boat, which has previously been heated in a blast flame and cooled in a desiccator. This boat is introduced into the combustion tube at a distance of not less than 60 mm. from the nearer asbestos board. After the tube has been swept out with a current of purified hydrogen, the absorption vessels are connected, the portion of the tube containing the charcoal then heated, and the heat subsequently extended very gradually to that part containing the silica boat. The hydrogen current is continued for about fifteen minutes from the time when full heat has been applied to the substance, and its rate is increased during the last ten minutes so as to drive over all the gaseous products of the reaction. At the end of the combustion (one and a half to one and three-quarter hours in all) the five tared absorption vessels are detached, and dry air (free from carbon dioxide) passed through them for about ten minutes, after which they are separated, allowed to stand for an hour, and repeatedly wiped with a cloth until constant in weight. In calculating the increase in weight in the tubes following the iodine pentoxide apparatus, a deduction of 50 per cent. is made for the oxygen derived from the iodine pentoxide, and not from the substance. The tabulated results show that the method is as accurate as the usual combustion method of estimating carbon and hydrogen. In the possible case of substances yielding stable compounds of carbon and hydrogen which would partially condense in the absorption vessels and vitiate the results, it would probably be practicable to eliminate such source of error by mixing a weighed quantity of a pure oxidising agent, such as potassium permanganate, with the substance in the boat. C. A. M.

Detection of [Commercial] Invert Sugar by Means of β-Naphthol. F. M. Litterscheid. (*Chem. Zeit.*, 1913, **37**, 321.)—The test proposed depends on the presence of traces of furfural derivatives in commercial invert sugar, and on the fact that these derivatives yield a red coloration when treated with β-naphthol and sulphuric acid. For the detection of invert sugar in honey, about 20 grms. of the sample are stirred with 10 c.c. of ether, the ethereal solution is decanted, and the extraction is repeated. The ethereal extracts are now, without filtration, transferred to a porcelain basin, and after the addition of a crystal of β-naphthol the ether is allowed to evaporate at the ordinary temperature. The residue is then treated with 5 c.c. of 88 to 90 per cent. sulphuric acid, the latter being added carefully so that it flows over the whole surface of the residue. Should the honey contain not less than 5 per cent. of commercial invert sugar, a Bordeaux-red or blue-violet coloration will develop within thirty minutes; the blue-violet coloration is usually noticed at the outer edge of the mixture. Natural honey yields a dirty yellow coloration with the test, a faint pink tint being noticed in many cases. The sulphuric acid employed should be chemically pure, and its concentration should be within the limits mentioned. W. P. S.

Influence of Ammonium Sulphate on the Polarimetric Estimation of Lactose. G. Jahnson-Blohm. (*Zeitsch. physiol. Chem.*, 1913, **83**, 441-444.)—The author finds that the presence of ammonium sulphate in quantity equal to that employed in E. Salkowski's method (ANALYST, 1912, **37**, 350) for determining lactose in milk produces a small decrease in the rotation. For a lactose content of 4 to 6 per cent. the results are 0·10 per cent. low, and the author recommends the application of this figure as a correction. E. W.

Method of Estimating Sugar. I. Bang. (*Biochem. Zeitsch.*, 1913, **49**, 1-19.)—A copper solution is prepared by dissolving 160 grms. potassium bicarbonate, 100 grms. potassium carbonate, and 66 grms. potassium chloride, in about 700 c.c. water, adding 4·4 grms. copper sulphate and diluting to 1 litre. Of this solution, 300 c.c. are diluted with a saturated potassium chloride solution to 1 litre. In preparing these solutions, it is necessary to avoid vigorous shaking, as this causes the absorption of too much air. Fifty-five c.c. of this solution are added to the sugar solution contained in a 100 c.c. Jena flask to the neck of which is attached a thick rubber tube. The mixture is boiled for three minutes, the flask then closed by means of a strong pinchcock, and immediately after closing removed from the flame and cooled in a stream of water. It is essential to avoid access of air during cooling, to prevent oxidation of reduced copper. When cold the flask is opened, starch solution added, and reduced copper estimated by titration with standard iodine of convenient strength ($\frac{N}{10}$ to $\frac{N}{100}$). The following reaction occurs :

$$CuCl + I + K_2CO_3 = CuCO_3 + KCl + KI.$$

Tables are given showing the quantities of $\frac{N}{100}$, $\frac{N}{25}$, and $\frac{N}{10}$ iodine which correspond with quantities of 1 to 10 mgrms. dextrose. It is claimed that the results agree well with those obtained polarimetrically, and that the new copper solution is superior to the older ones in not being so readily reduced by substances other than sugar, which is of special advantage in determining sugar in blood and urine. E. W.

Estimation of Sugars by the Kjeldahl-Bertrand Method. P. Beyersdorfer. (*Zeits. ges. Brauw.*, 1912, **35**, 556-559, 569-572, 582-585; through *Chem. Zentralbl.*, 1913, I., 1066-1067.)—According to Kjeldahl, two different sugars may be estimated in presence of each other by determining the reducing power of different quantities of the sugar solution towards different quantities of Fehling's solution. Such estimation, however, is not in fact possible. The cupric reducing power is not an exact additive function in the sense assumed by Kjeldahl, and the solution of a mixture of sugars does not behave towards different quantities of Fehling's solution as a solution of a single sugar. This principle may be applied for the qualitative detection of a mixture of two sugars. If with the same quantity of the solution to be examined different cupric reducing values be found with different quantities of Fehling's solution, the substance is a mixture of two or more reducing bodies. From the optical activity and the dry substance of a solution containing only two sugars, the quantity of each sugar may be calculated approximately, but this is not valid if a third substance which is inactive be also present. In such cases the cupric reducing value of the solution—which, however, must not contain more than 1 per cent. of reducing substance—affords the necessary data.

For the hydrolysis of beer extract the author employs, instead of hydrochloric acid, a solution of sulphuric acid (1 : 3), heating the mixture for five to six hours; the acid may then be easily removed by barium carbonate, and the hydrolysed products fermented by yeast. Nevertheless the barium carbonate acts upon the dextrose, possibly on account of its alkaline reaction, and this action has an influence on the optical rotation of the inverted extract. The fermentable sugar in wort and beer could not be estimated by the quantity of carbon dioxide evolved on fermentation, owing to experimental difficulties; it was determined from the specific gravity before and after fermentation. J. F. B.

Estimation of Reducing Sugars by Lehmann's Method. L. Grimbert. (*Bull. Soc. Chim.*, 1913, **13**, 117-120.)—The various modifications of Lehmann's method (iodimetric estimation of copper remaining in solution after treatment with excess of Fehling's solution) are discussed, and that of Bertrand (*Bull. Soc. Chim.*, 1906, **35**, 1285) is recommended. The use of Bertrand's copper solution and alkali tartrate solution (*loc. cit.*) are recommended. The $\frac{N}{10}$ iodine solution is standardised by means of arsenious acid, and the thiosulphate solution is adjusted to be exactly equivalent to the iodine solution under the conditions obtaining in a titration of the acidified filtrate from a copper reduction experiment—that is to say, in presence of 50 c.c. of 6 per cent. sulphuric acid. The copper solution is standardised by taking 20 c.c. together with 20 c.c. of the alkali tartrate solution, diluting to 200 c.c., withdrawing a portion of 50 c.c., adding 8 c.c. of dilute (1 : 1) sulphuric acid and 10 c.c. of 20 per cent. potassium iodide. After a few minutes 10 c.c. of thiosulphate solution is added, and the excess of thiosulphate titrated with iodine in presence of starch solution. In estimating reducing sugars, one takes 20 c.c. each of copper solution and alkali tartrate solution, not more than 0·1 grm. of sugar, and enough water to make the total volume 60 c.c., and boils for exactly three minutes. The mixture is cooled, made up to 200 c.c., filtered, and 50 c.c. of the filtrate is acidified, treated with potassium iodide, and titrated as already described. The difference between the volume of iodine solution required in the final titration and that required in standardising the copper solution is the measure of the sugar present, and its amount is given directly by reference to Bertrand's tables (*loc. cit.*). The author has checked many of Bertrand's figures for dextrose, and in no case do his numbers differ from those of the earlier author by as much as 1 per cent. G. C. J.

Accuracy of the Estimation of *l*. Tyrosine in Proteins. E. Abderhalden and D. Fuchs. (*Zeitsch. physiol. Chem.*, 1913, **83**, 468-473.)—The colorimetric method for determining tyrosine described by O. Folin and W. Denis (*J. Biol. Chem.*, 1912, **12**, 245) yields results which are too high because similar colours are produced by oxytryptophane and tryptophane. The authors agree with Folin and Denis in finding it frequently exceedingly difficult to separate quantitatively tyrosine by simple crystallisation. The cause of this has been traced in certain cases to absorption of acid or ammonia fumes from the atmosphere during evaporation in open basins. Much better results are obtained if the evaporation be conducted under reduced pressure. A more interesting reason for quite considerable quantities of tyrosine failing to crystallise is the formation of salt-like compounds with the basic constituents of proteins, such as the compound of tyrosine with lysine observed by E. Fischer and E. Abderhalden. In view of this experience, tests were made to determine whether it is possible to quantitatively recover tyrosine added to gelatine. Tyrosine-free gelatine was dissolved in five times its weight of 25 per cent. sulphuric acid, a known quantity of tyrosine added, and boiled for twenty hours. Baryta was then added to remove the sulphuric acid, and the barium sulphate extracted with boiling water until the extract no longer gave a violet colour with a 1 per cent. aqueous solution of triketohydrindenehydrate (Ninhydrin). This reagent proved much more delicate than Millon's reagent. By concentrating the combined filtrates at 45° C., it was possible to recover as much as 90 per cent. of the added tyrosine; in certain cases, however, the yield amounted to only 60 to 70 per cent. It was found possible to recover the tyrosine almost quantitatively by the following method: The liquid after hydrolysis is diluted until it contains 2·5 per cent. sulphuric acid. A 10 per cent. solution of phosphotungstic acid is then added with constant stirring, great care being taken to avoid the addition of any excess. The precipitate is filtered off and washed, and the filtrate treated with baryta to remove phosphotungstic acid and filtered. To this filtrate the exact amount of sulphuric acid needed to remove the excess of baryta is added and the barium sulphate extracted with boiling water until no coloration is given by "Ninhydrin." The filtrate was then concentrated at 40° C. under reduced pressure until the mother-liquor gave no reaction with Millon's reagent. The crude tyrosine which separated was purified by boiling with animal charcoal and fractional crystallisation from water until a sample on analysis yielded figures agreeing with pure tyrosine. E. W.

INORGANIC ANALYSIS.

Analysis of Aluminium Alloys. W. E. von John. (*Chem. Zeit.*, 1913, **37**, 363.)—In the analysis of alloys in which aluminium predominates, the separation of the aluminium is often tedious and apt to be attended by loss of the minor constituents. The following method of analysis is recommended as simple and yet capable of yielding results of a satisfactory degree of accuracy: The drillings (1 grm.) are dissolved in hydrochloric acid, and the usual trace of silica separated by evaporation to dryness. In the filtrate from the silica, lead and tin, if present, are separated by means of sulphuric acid and nitric acid. To the filtrate—now free from silica, lead, and tin—bromine water, and then caustic alkali are added. The mixture is heated to boiling, let stand overnight, and then filtered. The precipitate, which contains all the copper, iron, manganese, and magnesium, is washed and dissolved in dilute sulphuric acid, the copper separated as sulphide, and the filtrate boiled to expel hydrogen sulphide, and treated with bromine to oxidise iron. The excess of bromine is expelled by boiling, and iron precipitated by ammonia, the manganese in the filtrate from the ferric hydroxide being precipitated by means of bromine or hydrogen peroxide. In the filtrate from manganese, the magnesium is precipitated as magnesium ammonium phosphate. The filtrate from copper, iron, manganese, and magnesium, which contains all the zinc as well as the aluminium, is treated with ammonium sulphide, and the zinc sulphide filtered off and ignited with sulphur in a current of hydrogen. The zinc sulphide is dissolved in hydrochloric acid, and the solution tested for the presence of aluminium, which, if present, must be precipitated as hydroxide, and its weight deducted from the weight previously recorded as due to zinc sulphide. The test numbers are good. G. C. J.

Detection of Arsenic by Means of Bettendorf's Reagent. L. W. Winkler. (*Zeitsch. angew. Chem.*, 1913, **26**, 143-144.)—Statements have been made that Bettendorf's reagent (stannous chloride) reduces sulphates on heating, and that, consequently, the solution to be tested for arsenic must not be warmed after the addition of the reagent if the solution contains sulphates or sulphuric acid. The author finds that this is not the case; no reduction occurs on heating the mixture, and the presence of sulphuric acid is even of advantage, as it acts as a dehydrating agent. The reagent is best prepared by dissolving 100 grms. of stannous chloride, $SnCl_2,2H_2O$ (the crystals should be quite transparent), in 1 litre of concentrated hydrochloric acid (36 to 38 per cent. HCl). Usually traces of arsenic are present in the acid, and cause the solution to become light brown in colour; if, however, about 1 grm. of finely powdered glass is added, and the whole is kept in a warm place for from twenty-four to forty-eight hours, the arsenic will be precipitated and the clear, colourless solution may be decanted and stored in stoppered bottles. In carrying out the test, the solution of the substance under examination (preferably in hydrochloric acid) is mixed in a test-tube with five times its volume of the reagent, the mixture is heated just to boiling, and then placed aside for thirty minutes. Arsenic may be detected in concentrated sulphuric acid by mixing 1 c.c. of the acid with 1 c.c. of water, and adding 10 c.c. of the reagent. The minimum quantity of arsenic which can be detected by the test is about 0·001 grm. of arsenic trioxide per litre of solution, when 2 c.c. of the latter are used for the test. W. P. S.

Factors influencing the Estimation of Arsenic in Soils. J. E. Greaves. (*J. Amer. Chem. Soc.*, 1913, **35**, 150-156.)—On account of its extensive use as an insecticide, arsenic may accumulate in soils to such an extent as to become injurious to vegetation; it also occurs in many virgin soils. The author confirms the work of previous investigators on the retarding effect which the presence of iron exerts upon the arsenic liberated by the Marsh method. Working with 1 mgrm. of arsenic, 1 mgrm. of iron, 25 grms. of zinc, and dilute sulphuric acid, it was found that one quarter of the amount of arsenic was retained. It is shown that the retarding action of 1 mgrm. of iron can be overcome by the use of 200 mgrms. of stannous chloride, and that for 0·5 grm. of iron 1 grm. of stannous chloride suffices. Since arsenic trichloride is volatile at comparatively low temperatures, tests were made with 10 grms. of soil, containing varying amounts of sodium chloride and 1 mgrm. of arsenic, in all cases using 2 grms. of stannous chloride. The result showed that no appreciable quantity of arsenic is lost, even when the percentage of chloride in the soil reaches 3 per cent. The method which has given the best results is as follows:

To 10 grms. of soil are added 25 c.c. of concentrated nitric acid and the mixture heated for thirty minutes. While still moderately hot, 10 c.c. of concentrated sulphuric acid are added, and the whole heated for thirty minutes. The soil is thoroughly washed with hot distilled water, the filtrate evaporated to dryness, and heated until free from nitrates. The residue is then treated with dilute arsenic-free sulphuric acid to which has been added 20 c.c. of 10 per cent. stannous chloride solution. The solution is slowly introduced into a Marsh apparatus, the glass tube through which the hydrogen was conducted being heated by means of an electric combustion furnace, the temperature of which was maintained just below the melting-point of hard glass by means of a rheostat. The tube and deposit of arsenic obtained after the apparatus has been running two hours is then carefully weighed on a fine analytical balance, the arsenic being dissolved out by dilute nitric acid, the tube washed in water, alcohol, and ether, dried, and then weighed as before.

H. F. E. H.

Determination of Copper in White Metal Alloys. F. Ibbotson and L. Aitchison. (*Chem. News*, 1913, **107**, 121.)—The drillings (0·5 grm.) are dissolved in 10 to 15 c.c. of *aqua regia*, an equal volume of water is added, and the solution cooled. If lead chloride separates, it is filtered off on a pulp filter, which is washed with cold water. About 1 grm. of tartaric acid is added, and, when this has dissolved, the solution is nearly neutralised with sodium hydroxide, heated to boiling and added, drop by drop from a separating funnel, to a boiling solution containing 20 grms. of sodium hydroxide and 2 to 3 grms of hydrazine hydrochloride in 250 to 300 c.c. of water. The latter solution is agitated vigorously during the operation. After digesting the mixture for about fifteen minutes, the precipitate of metallic copper is filtered off on a filter of ashless paper pulp, washed with hot water, dissolved on the filter by means of dilute nitric acid (sp. gr., 1·1), the filter washed, and the filtrate of copper nitrate evaporated to pastiness. On taking up in water and a few drops of nitric acid, it should be free from any opalescence which would be caused by unseparated stannic oxide, and the weight of the filter ash should be negligible. Sodium carbonate solution is next added to the copper solution until it is alkaline, then acetic acid in slight excess, and the solution is boiled. When cold, potassium iodide is added, and the liberated iodine is titrated with thiosulphate, of which 1 c.c. = 1 mgrm. copper. The method is accurate with alloys containing 80 per cent. of lead, together with tin and antimony, and also with alloys in which tin predominates. The small quantities of zinc which occur in these alloys are completely separated from copper, and the minute quantities of iron unavoidably present in white metals are without appreciable effect on the results. G. C. J.

Electrolytic Estimation of Copper in Ores containing Arsenic, Antimony, or Bismuth. D. J. Demorest. (*J. Ind. and Eng. Chem.*, 1913, **5**, 216.) —The following method is less tedious than methods in which the metals are first precipitated as sulphides, arsenic and antimony removed by means of sodium sulphide, and copper separated from bismuth by means of cyanide. One grm., or more if the sample is low in copper, is decomposed by heating with 7 c.c. of hydrochloric acid and 7 c.c. of nitric acid, and the mixture is evaporated with 5 c.c. of sulphuric acid until it fumes. About 30 c.c. of 10 per cent. tartaric acid solution is added, the mixture heated till all soluble matter is dissolved, allowed to cool somewhat, made alkaline with ammonia and then acid with sulphuric acid, of which about 1 c.c. more is used than is necessary to neutralise the ammonia. To the hot solution, 2 grms. of sodium sulphite is added, and the mixture stirred until solution is complete. Then a strong solution of potassium thiocyanate, containing about 1 grm. of the salt, is added, the solution stirred vigorously, heated to boiling, and the precipitate given a few minutes to subside. The precipitate is filtered off on a paper of close texture, and washed several times with a solution containing 1 per cent. of thiocyanate and 1 per cent. of tartaric acid. It may then be ignited with the paper, dissolved in 5 c.c. of nitric acid, diluted and electrolysed, or, preferably, it is dissolved on the filter with 18 c.c. of dilute (1:2) nitric acid, the funnel being covered to avoid loss, the filtrate is heated to boiling, and passed once more through the filter, which is washed with about 100 c.c. of water, and the filtrate and washings are boiled vigorously for five minutes to destroy thiocyanic acid, the bulk adjusted to about 125 c.c., and the solution electrolysed, using a gauze cathode, a drop of potential of 3 volts and a current of 3 ampères. Precipitation is complete in thirty to sixty minutes, and the deposit is firmly adherent if the boiling to destroy thiocyanic acid was sufficiently prolonged. Copper (0·15 grm.) may be determined in presence of an equal weight of arsenic, antimony, bismuth, iron, and lead, with an error not exceeding − 0·5 mgrm. The test numbers show no example of over-estimation of copper. G. C. J.

Thiocyanate-Permanganate Method for Copper in Ores. D. J. Demorest. (*J. Ind. and Eng. Chem.*, 1913, **5**, 215-216.)—In Guess's method for the determination of copper, the cuprous thiocyanate is decomposed on the filter by means of sodium hydroxide, and the resulting solution of sodium thiocyanate is acidified and titrated with permanganate. Theoretically $5Cu = 5HCNS = 6KMnO_4$, but under the conditions prescribed by Guess the reaction does not proceed quantitatively, and an empirical factor (about 1·05 the theoretical) has to be determined by the operator (see Low's "Technical Methods of Ore Analysis"; also Grossmann and Holter, ANALYST, 1909, **34**, 244). The reaction may be made approximately quantitative and the use of an empirical factor dispensed with by adding the bulk of the permanganate to the alkaline solution and only adding sulphuric acid when the thiocyanate reaction has nearly disappeared. The ore is heated for several minutes with 5 c.c. of strong hydrochloric acid, 10 c.c. of nitric acid are then added, and the digestion continued until the ore is completely decomposed. After addition of 10 c.c. of dilute (1 : 1) sulphuric acid, the mixture is evaporated until it fumes, allowed to cool, and then warmed with 50 c.c. of 6 per cent. tartaric acid solution until all soluble salts are dissolved. Ammonia is added until the appearance of a deep blue colour, then sulphuric acid until the reaction is acid, and then a further 1 c.c. of acid. After addition of 20 c.c. of 5 per cent. sodium sulphite solution, the mixture is heated nearly to boiling, and 20 c.c. of 5 per cent. potassium thiocyanate is added slowly with vigorous stirring. The mixture is maintained at a temperature near the boiling-point for several minutes, allowed to cool somewhat, and then filtered through asbestos contained in a Gooch crucible. The precipitate is washed with water, the pump-flask changed, and 30 c.c. of hot 10 per cent. sodium hydroxide poured through the filter, which is washed with water. The filtrate is heated to about 50° C. and permanganate run in from a burette until a drop of the solution, when tested on a white plate with a drop of a strongly acid solution of ferric chloride, shows only a faint coloration. About 30 c.c. of dilute sulphuric acid are then added to the contents of the titration flask, which are shaken until the manganese dioxide all dissolves, and the titration is completed in acid solution. If only about 1 c.c. of permanganate is required at this stage, the error of the method does not exceed 1 per cent. No element, except silver, interferes. G. C. J.

Electrolytic Separation of Copper from Tungsten and Molybdenum. W. D. Treadwell. (*Zeitsch. Electrochem.*, 1913, **19**, 219-221.)—The use of potassium cyanide to effect this separation is objectionable, and its effectiveness in the case of molybdenum doubtful. Copper may be readily separated from tungsten as follows: The solution (150 c.c.), containing the tungsten as alkali tungstate and the copper as chloride, nitrate, sulphate, phosphate, or acetate, is made strongly alkaline by addition of 15 to 20 c.c. of strong ammonia, and 3 to 5 grms. of ammonium sulphate are also added. The solution is then electrolysed by connecting the electrodes direct to the terminals of a single lead accumulator. As much as 0·2 grm. of copper can be deposited in three hours, or in twenty minutes if a rotating electrode be employed. As the results are unaffected by leaving the cell connected to the accumulator after all the copper is out of solution, it is convenient when using stationary electrodes to allow the electrolysis to proceed overnight.

The above process cannot be applied without modification to the separation of copper from molybdenum, since, at 1·8 to 2 volts, alkali molybdates are reduced to salts of molybdenum, and ultimately molybdenum hydroxide is deposited on the cathode. If, however, the difference of potential between the terminals be not allowed to exceed 0·9 volt, if the deposition of the copper be facilitated by the addition of a suitable reducing agent, and if certain precautions be taken, a quantitative separation of these metals is possible. The reducing agent recommended is sodium bisulphite, at the rate of 15 to 30 c.c. of the commercial solution to 150 c.c. of electrolyte, to which 15 to 20 c.c. of ammonia is also added. Agitation of the electrolyte is essential to success, the cathode deposit must remain covered with liquid to avoid oxidation of the copper, and the current must be broken without delay when all the copper is out of solution. The rapid falling off of the current indicates the completion of the deposition, but, if there is no ammeter in the circuit, it suffices to break the current five minutes after the solution has ceased to possess a perceptible blue colour. As much as 0·25 grm. copper can be deposited in thirty minutes with an error not exceeding ± 0·5 mgrm. G. C. J.

Estimation of Hypochlorites. H. G. Williams. (*Chem. News*, 1913, **107**, 109.)—Roncali and Roberts (*Chem. Central*, 1904, 1, 1294) suggest the estimation of hypochlorites by adding (slowly) a known volume of the hypochlorite solution to a boiling solution of hydrazine sulphate in dilute sulphuric acid and measuring the volume of nitrogen liberated, "the available chlorine" being double this. The following method is simpler and sufficiently accurate:

The hypochlorite is titrated with a solution of hydrazine sulphate containing 3·2535 grms. per litre (each c.c. = 0·0008 grm. of oxygen = 0·003546 grm. of chlorine), the end-point being determined by iodide-starch paper. If the alkali always present with hypochlorites is not sufficient to neutralise the sulphuric acid liberated, sodium bicarbonate in sufficient quantity must be added. The action appears to be—

$$N_2H_4 + 2(MOCl) = N_2 + 2H_2O + 2(MCl).$$

Results are recorded in which sodium hypochlorite and bleaching powder are tested by the above method against a titration with $\frac{N}{10}$ arsenious acid, the average difference with the hypochlorite being 0·075 per cent., and with the bleaching powder 0·11 per cent. A solution of hydrazine sulphate is much easier to prepare than $\frac{N}{10}$ As_4O_6, and appears to keep better.

Browne and Shetterly (*J. Chem. Amer. Chem. Soc.*, 1908, **30**, 53) find that when hydrazine sulphate is oxidised by chlorates or by free chlorine, ammonia and hydrazoic acid are formed, but no trace of these can be detected in the mixtures after titration (*cf.* ANALYST, 1908, **33**, 24). H. F. E. H.

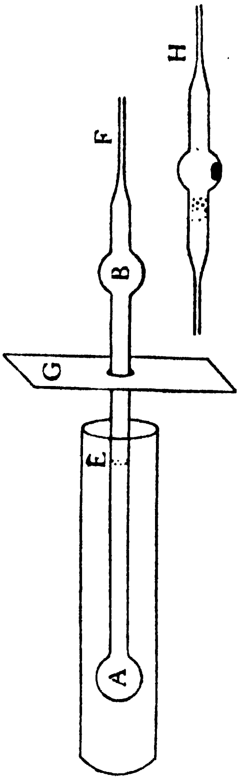
Estimation of Manganese in the Presence of Iron. M. E. Pozzi-Escot. (*Ann. Chim. anal.*, 1913, **18**, 56-58.)—An iodimetric method is recommended. The solution containing iron and manganese is heated to boiling and treated with an excess of sodium hydroxide and about 1 grm. of sodium persulphate; the precipitated hydroxides are washed by decantation, then collected on a small filter, and washed until the filtrate is neutral. The filter and precipitate are now transferred to a flask of about 200 c.c. capacity, the neck of which is closed with a hollow ground-in stopper. This stopper is provided with a side tube which is extended to form a series of five absorption bulbs, and a second tube passes through the stopper to nearly the bottom of the flask. Twenty c.c. of concentrated hydrochloric acid are then introduced into the flask and the contents are boiled, the liberated chlorine passing through the absorption bulbs, in which has been placed previously a solution containing 3 grms. of potassium iodide and 5 grms. of sodium acetate, together with 5 grms. of zinc oxide in suspension. A current of carbon dioxide is passed through the apparatus during the boiling, which should not be prolonged for more than a few minutes, and the liberated iodide in the bulbs is then titrated with $\frac{N}{10}$ thiosulphate solution. Each c.c. of the latter solution is equivalent to 0·00275 grm. of manganese. W. P. S.

Colorimetric Estimation of Small Quantities of Manganese in Water. F. Haas. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1913, **25**, 392-395.)—With slight modification, the persulphate method described by Marshall (ANALYST, 1901, **26**, 195) yields accurate results. The alterations recommended consist in treating the water directly with sulphuric acid and persulphate without the addition of nitric acid and silver nitrate, as this addition serves no good purpose and actually interferes with the test. One hundred c.c. of the water to be tested are acidified with 5 c.c. of 20 per cent. sulphuric acid, from 0·5 to 1 grm. of solid potassium persulphate is added, and the mixture is heated slowly until either a red-violet coloration develops or a brown colour, due to separated manganese peroxide, is obtained. The mixture is now cooled, a trace of sodium hydrogen sulphite is added, and the solution again heated with persulphate until the permanganate coloration reaches its full intensity. After cooling, the coloration is compared with that shown by known quantities of permanganate. The most trustworthy results are obtained with quantities of manganese varying from 0·05 to 1·0 mgrm. per litre of water. W. P. S.

Estimation of Small Quantities of Manganese and Chromium in Minerals and Rocks. M. Dittrich. (*Zeitsch. anorg. Chem.*, 1913, **80**, 171-174.)—In rocks containing traces of chromium of the same order of magnitude as the manganese, the colorimetric determination of the latter is impossible unless a separation of the two metals is effected. This may be easily accomplished as follows: The rock is opened up with hydrofluoric and sulphuric acids and treated with persulphate and silver nitrate in the usual manner. Excess of ammonia is next added, and the solution warmed for a short time, when all the manganese comes down with the iron, whereas the chromate is not reduced. The precipitate is filtered off, washed, dissolved by means of sulphuric acid and sulphurous acid (or hydrogen peroxide), the excess of the reducing agent removed or destroyed by boiling, and the treatment with persulphate and silver nitrate repeated. The chromium in the filtrate may be estimated by freeing the latter from silver, concentrating to small bulk, and comparing colorimetrically with a standard solution of potassium chromate. G. C. J.

Separation of Niobium and Tantalum by Means of Potassium Chloride in Hydrofluoric Acid Solution. Meimberg and P. Winzer. (*Zeitsch. angew. Chem.*, 1913, **26**, 157-158.)—After describing their failure to obtain a satisfactory separation by other methods, the authors recommend the following: From 5 to 10 grms. of the mixed acid earths are dissolved in hydrofluoric acid, of which enough must be taken to allow the whole of the tantalum and niobium to form potassium double fluorides on the subsequent addition of excess of potassium chloride. Excess of a saturated solution of the latter salt is next added, and the solution allowed to cool. As the double fluoride of tantalum and potassium is not quite insoluble in a fairly concentrated solution of potassium chloride, the bulk of the solution is kept as small as possible, but there must be at least 12 c.c. of liquid for every gram of potassium niobium fluoride to be separated. The tantalum double salt is filtered off, washed with a cold saturated solution of potassium chloride, and reconverted into tantalic acid by digestion with hot sulphuric acid. The acid is again dissolved in hydrofluoric acid, and separated from traces of niobium by reprecipitation as double fluoride in the manner already described. The bulk of the solution may be kept smaller this time, as only traces of niobium have to be removed. The double salt is filtered off, washed with potassium chloride solution, decomposed by digestion with hot sulphuric acid, and the residue boiled with water containing hydrochloric acid, and then with ammonia, the separated tantalic acid (and titanium dioxide if present in the original material) being filtered off, ignited, and weighed. Titanium, if present, is estimated colorimetrically, and a correction applied to the weight of the mixed oxides. An alternative method is to carry the process described above as far as the first separation of the double fluoride, and to separate the last traces of niobium by solution in hydrofluoric acid and treatment with potassium chloride solution. After allowing to cool, the purified double salt is filtered off, washed, and converted into tantalic acid in the manner already described. No test numbers are given, but the method is said to be applicable to the technical production of tantalum compounds free from all but a negligible trace of niobium. G. C. J.

Estimation of Mercury as Metal by the Dry Method. A. C. Cumming and J. Macleod. (*J. Chem. Soc.*, 1913, **103**, 513-516.)—The use of Penfield's apparatus for water estimations obviates the experimental difficulties in the estimation of mercury by the dry method. It consists of a tube about 15 cm. long and 5 cm. in diameter, closed at one end by a bulb *A*, and with a second bulb blown at *B*. The dried tube is weighed, a suitable quantity of the substance containing mercury introduced through a long funnel into the bulb *A*, and the tube and its contents again weighed. A mixture of iron filings and quicklime is next added by means of the funnel, and the whole contents of the bulb thoroughly mixed by rotating the tube, after which a mixture in about equal parts of iron filings and quicklime is introduced so as to fill about 8 cm. of the tube. This is kept in position by means of an asbestos plug at *E*, a shallow air passage being left at the top of the powder by tapping the tube. Finally, the open end of the tube is drawn out to a capillary as shown at *F*, and the whole tube enclosed in an iron tube about 15 cm. by 1·5 cm., the bulb *A* being first wrapped in asbestos-paper. The ends of the iron tube are closed with asbestos, and an asbestos shield fixed at *G* to prevent the bulb *B* from becoming hot. This bulb is also cooled by means of wet filter-paper. The iron tube is first heated with a small flame at the end near *G*, and the heat gradually increased and extended until the whole of the tube is of a dull red heat. After the whole of the mercury has distilled and has been collected in *B* (about an hour), the glass tube is withdrawn from the iron tube and heated at a point about midway between *E* and *B*, so that it can be drawn out into a tube as shown at *H*. Any moisture is removed by drawing a current of dry air through this tube until the weight becomes constant, after which the mercury is expelled by shaking the tube, and if necessary heating it and blowing air through it. Finally, it is cooled and again weighed. The method is shown to give accurate results, but in the case of mercuric iodide and mercuric sulphide, it was necessary to add lead chromate to the mixture in the proportion of 1 part to 2 of iron filings and 1 of quicklime. For the estimation of mercury in minerals of unknown composition from 0·5 to 1 grm. of the sample should be heated, as described, with 2·5 to 3 grms. of the mixture containing the lead chromate. C. A. M.



Application of Selenic Acid to the Estimation of Bromide in Presence of Chloride. F. A. Gooch and P. L. Blumenthal. (*Zeitsch. anorg. Chem.*, 1913, **80**, 161-170.)—Selenic acid is capable of reacting with bromides and chlorides, with liberation of the halogen and formation of selenious acid, but the reaction in either case is a reversible one. The present paper describes researches directed to the determination of the conditions under which the reaction with bromide is approximately quantitative, and that with chloride almost wholly inhibited. In mixtures of 0·1 grm. potassium bromide with 0·1 grm. sodium chloride, the bromine may be estimated as follows, with an error of $-0{\cdot}6 \pm 0{\cdot}5$ mgrm.: The solution (30 c.c. or more) is contained in a flask fitted with a tap funnel and a gas exit tube. The tap funnel is charged with 5 c.c. of 40 per cent. selenic acid, or with 2 grms. of sodium selenate dissolved in 3 c.c. of dilute (1 : 1) sulphuric acid, and its constricted top is connected to a carbon dioxide generator. The gas exit tube is bent, and dips into 10 to 15 c.c. of water containing 2·5 c.c. of selenic acid (or 0·3 grm. selenate and 1 to 2 c.c. of dilute sulphuric acid), and contained in a flask which is maintained at 115° to 120° C. in an oil-bath. This apparatus serves to oxidise any hydrogen bromide which may escape oxidation in the reaction flask proper. The oil-bath being hot, the selenic acid is allowed to flow into the reaction flask, and a current of carbon dioxide is passed through the apparatus, whilst the contents of the reaction flask are heated to boiling and concentrated to about 10 to 15 c.c. The gases passing away from the second flask are conducted below the surface of 200 c.c. of water containing in solution 3 or 4 grms. of potassium iodide, and the usual precautions are taken to guard against loss of iodine. The liberated iodine is titrated, and its bromine equivalent calculated in the usual manner. If the concentration is not pushed below 10 c.c., no chloride is decomposed. The oxidation of the bromide is never quite complete, but the results are fairly concordant, being, as stated above, from 0·1 to 1·1 mgrms. low. G. C. J.

Gay-Lussac Method of Silver Determination. F. P. Dewey. (*J. Ind. and Eng. Chem.*, 1913, **5**, 209-214.)—The exact modification of the method employed in the United States mint service is described, and the results of some thousands of assays are tabulated to show that two operators, working on identical samples of standard silver and making four determinations each, may differ as much as 1 fine in their reports. Nevertheless, the method is preferred to fire assay for mint work. It is useless for a person who uses it only occasionally to expect to attain high accuracy with it, without spending more time upon a determination than is warranted in a busy commercial laboratory. G. C. J.

Volumetric Method for the Estimation of Sulphur in Illuminating Gas. F. Schulz. (*Petroleum*, 1913, **8**, 585-586; through *Chem. Zentralbl.*, 1913, I., 1063.)—As a technical method, only the lamp method of Heusler and Engler is generally available, that of Hempel and Graefe being not quite satisfactory with volatile oils. The lamp method may be still further simplified by passing the gaseous products of combustion through a solution of hydrogen peroxide, and estimating the sulphuric acid produced. The absorption flask is charged with 30 c.c. of a 3 per cent. solution of hydrogen peroxide, diluted with 30 c.c. of water, and neutralised to methyl orange with $\frac{N}{10}$ sodium hydroxide. In the case of petroleum, 5 to 6 grms. are burnt (at the rate of 1·3 to 1·6 grms. per hour). A strong current of air is drawn through the apparatus to insure freedom from smoke, otherwise the absorption would be incomplete. The estimation of sulphur dioxide in the air used for combustion is carried out in a blank experiment. After weighing back the lamp, the acidity produced in the absorption liquid is titrated with $\frac{N}{10}$ alkali. If the influence of oxides of nitrogen is to be eliminated, hydrogen peroxide free from sulphuric acid should be used for the absorption. According to the author's determinations, samples of European petroleum contained from 0·056 to 0·077 per cent. of sulphur. J. F. B.

Volumetric Method for the Estimation of Tin by Means of Potassium Bromate. F. Fichter and E. Müller. (*Chem. Zeit.*, 1913, **37**, 309.)—The following method, due to Zschokke, but hitherto unpublished, has proved of special value in silk works, and in the author's hands gives more accurate results than they are able to get by the iodimetric method. The tin solution (20 c.c.), which should be approximately $\frac{N}{10}$ and not too strongly acid, is treated in the cold with about 0·15 grm. of aluminium in the form of wire. The separated tin is next dissolved by adding to the contents of the flask 30 c.c. of hydrochloric acid and 20 c.c. of water, and boiling, oxidation of the stannous chloride being prevented by the use of a Bunsen valve. The cooled solution is then titrated with $\frac{N}{10}$ potassium bromate until the appearance of the yellow colour due to the bromine, which results from the interaction of hydrogen bromide with the first drop in excess of the reagent. The bromate solution is standardised iodimetrically. For use in silkworks, the method has the advantage that it is available in presence of phosphates. G. C. J.

Estimation of Titanium in Presence of Iron. W. M. Thornton jun. (*Chem. News*, 1913, **107**, 121-122.)—Gooch's method for the gravimetric estimation of titanium in presence of iron, aluminium, and phosphoric acid, leaves nothing to be desired in point of accuracy, but is unnecessarily tedious for the analysis of minerals and rocks which contain little or no aluminium, but much titanium. In the analysis of such materials the author proceeds as follows: Tartaric acid is added to the solution in amount equal to at least three times the weight of the oxides of titanium and iron, and the iron is next reduced to the ferrous state by means of hydrogen sulphide. The solution is then rendered faintly alkaline with ammonia, and hydrogen sulphide is again passed in until precipitation of the iron is complete. The ferrous sulphide is filtered off and washed with very dilute, colourless ammonium sulphide, and the titanium in the iron-free filtrate is determined as follows: The filtrate is evaporated with 10 to 15 c.c. of sulphuric acid until the tartaric acid begins to char. After cooling a little, 5 c.c. of fuming nitric acid are added cautiously, and the decomposition of the tartaric acid completed by the cautious application of heat until fumes of sulphuric acid finally appear. The liquid is diluted by pouring it into 100 c.c. of water, and ammonia is added until the appearance of a precipitate which disappears with difficulty. About 1 c.c. of a strong solution of ammonium bisulphite is added to hold up any trace of iron which may remain in the solution, and this is followed by 5 c.c. of acetic acid and 15 grms. of ammonium acetate. The solution is then diluted to about 350 c.c., heated to boiling, and maintained in ebullition for two minutes. The titanium hydroxide, which comes down in an easily filterable condition, is filtered off, washed successively with very dilute acetic acid and water, and finally ignited for twenty minutes over a Meker burner. Titanium dioxide (0·13 grm.) can be separated from an equal weight of ferric oxide with an error not exceeding +0·6 mgrm., and the titanium is never under-estimated. G. C. J.

New Method for Volumetric Estimation of Uranium. V. Auger. (*Bull. Soc. Chim.*, 1913, **13**, 109-117.)—In presence of iron, but absence of titanium, uranium may be estimated by reduction of the acid solution by means of amalgamated zinc, and titration of the quadrivalent uranium by means of a standard ferric solution, which oxidises it to the hexavalent condition, thiocyanate serving as indicator. As the concentration of acid is increased, a higher temperature is necessary to bring about the reaction with the ferric salt, and at higher temperatures the error due to oxidation by the air ceases to be negligible. For this reason, and because high saline concentrations exercise a prejudicial influence, though large quantities of salts are without effect unless their concentration is very high, the reduced solution is diluted considerably before titration. Hydrochloric acid should not be used for acidifying the solution to be reduced, since aerial oxidation at a later stage is more to be feared in presence of much hydrochloric acid; but chlorides in considerable quantity are without effect. The ferric solution, which may conveniently be $\frac{N}{10}$ or $\frac{N}{50}$, is preferably a solution of iron alum containing free sulphuric acid. It is standardised by means of a solution of uranium of known strength. The solution to be analysed, or 25 c.c. of the standard uranium solution, is rendered strongly acid by addition of 25 to 50 c.c. of dilute sulphuric acid (1 : 10), and the mixture is poured into a flask nearly filled with amalgamated zinc. At the temperature of the water-bath reduction is complete in ten minutes. The cooled solution is filtered through a plug of glass-wool, the zinc is washed with acidulated water, and the filtrate and washings are aerated by shaking or the use of a current of air to oxidise any trivalent uranium which may possibly be present. Ammonium thiocyanate solution, equivalent to 2 grms. of the salt, is added, and the mixture diluted to 200 c.c. or more with hot water. The titration with ferric solution is then carried out at a temperature of 40° to 50° C.

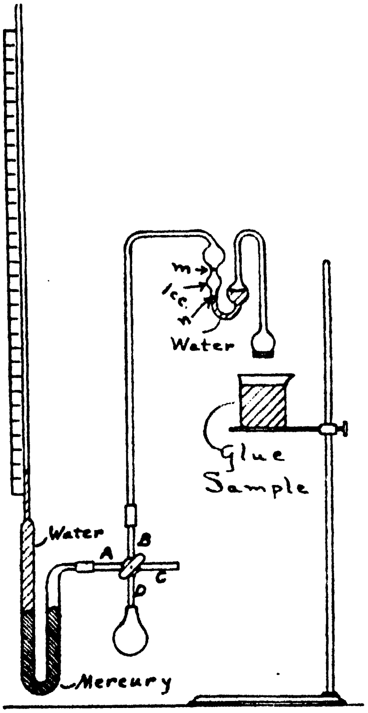
The above method is not available in presence of titanium. In presence of titanium and iron, the following method is recommended. It depends on the reduction of the uranium and iron in alkaline tartrate solution by means of titanous sulphate, using azo-induline to determine the presence of the least possible excess of the reagent, followed by titration of the uranium with ferric sulphate in acid solution in the manner already described. The titanous solution is made by dissolving 100 grms. of hydrated titanium dioxide in 300 grms. of hot, slightly diluted sulphuric acid, diluting to 1,000 c.c., and reducing in the cold with amalgamated zinc. A weaker titanous solution is also required, and is made by diluting some of the stronger solution to one-tenth strength. The tartrate solution is made by saturating 150 grms. of tartaric acid with sodium carbonate and diluting to 500 c.c. The reduction is effected in a flask with a wide neck, fitted with a double-bored stopper, one hole of which serves for the introduction of a current of carbon dioxide, whilst the other carries a straight tube 5 cm. long and as wide as a test-tube.

Enough tartrate solution to saturate any free mineral acid and a few drops of a 1 per cent. solution of azo-induline are added, and titanous chloride is then run in until the violet colour gives place to orange, using the stronger titanous solution until approaching the end-point if the sample contains large quantities of iron and only little uranium. If the end-point be overstepped, ferric solution is added until the colour changes, and then again dilute titanous solution, drop by drop. Enough hydrochloric acid is now added to insure the presence of free mineral acid (2 c.c. of acid to each 5 c.c. of tartrate solution taken), and the uranium is finally titrated with ferric solution, as already described. G. C. J.

Radio-Activity of Solutions of Uranium Salts: Method for the Estimation of Uranium. L. Michiels. (*Bull. Soc. Chim. Belg.*, 1913, **27**, 69.)—Solutions of uranium salts were found to give α-ray standards of greater accuracy than can be obtained by the use of the powdered solid substance. The ionisation produced is, moreover, a measure of the amount of uranium present. A series of solutions of potassium uranyl sulphate and of uranyl nitrate were examined by means of a special electroscope, the amber insulation of which was unaffected by an atmosphere saturated with moisture. With constant volume, area, temperature, and depth of solution, the α-radiation was proportional to the quantity of uranium in the solution. With concentrated solutions, however, the ionisation did not keep pace with the increase in concentration, but if, however, a series of solutions of the uranium salt of different concentrations are brought to the same sp. gr. by means of ferric chloride, the proportionality between ionisation and concentration is restored. It is thus possible, in the absence of other radio-active substances, to estimate uranium without preliminary separation from other metals.
O. E. M.

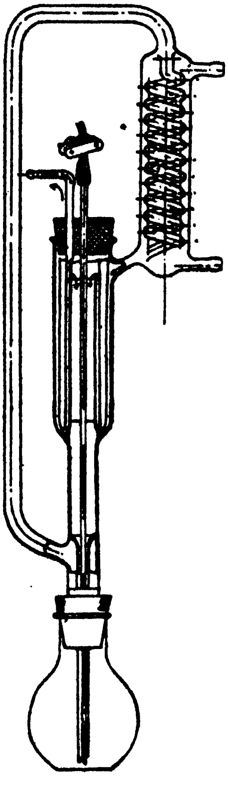
APPARATUS, ETC.

Improved Apparatus for Testing the Jelly-Strength of Glues. E. C. Hulbert. (*J. Ind. and Eng. Chem.*, 1913, **5**, 235.)—The apparatus consists of a thistle-tube, the stem of which is twice bent as shown. The two larger bulbs are 2 cm. in diameter, and serve as safety-traps; the smallest is graduated to contain exactly 1 c.c. Over the mouth of the thistle-tube is stretched a diaphragm of thin rubber. The far end of this tube connects through a 4-way stopcock with a mercury-filled U-tube, having its further arm extended upwards and of smaller bore, containing water or other liquid above the mercury to serve as a gauge. A rubber bulb is attached to one arm of the stopcock, the fourth arm being left open to the air. The sample of glue-jelly is raised by means



of the adjustable stand until the liquid in the bulb-tube reaches the upper graduation; the stand is then secured, and the stopcock set to connect the arms *A*, *B*, and *C*. The cock is next turned to connect the arms *A*, *B*, and *D*, and enough air is forced into the system by the rubber bulb to force the liquid in the bulb-tube down to the lower graduation, the cock being then turned to an intermediate position and the reading taken. The reading may be repeated several times on the same sample, and is uninfluenced by the condition of the surface of the sample, such as skin formation. The error due to supporting a column of water resting on the glue surface, as in some similar apparatuses, is avoided; this is important in cases of low-grade glues.

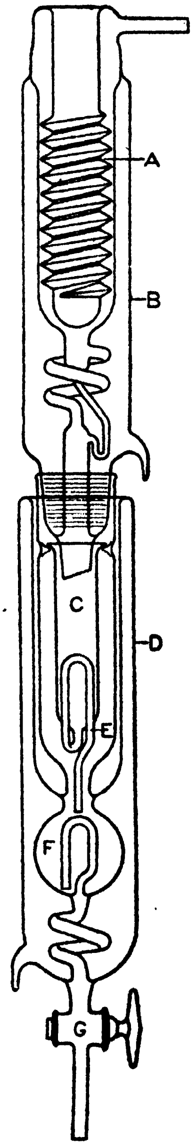
H. F. E. H.

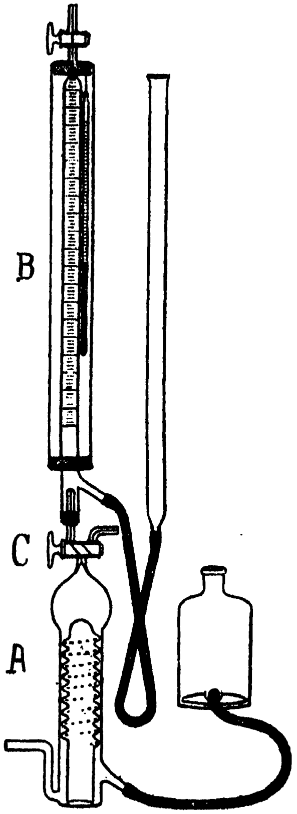


New Apparatus for Quantitative Dialysis. A. Golodetz. (*Chem. Zeit.*, 1913, **37**, 259-260.)—The principle of dialysis has hitherto been but rarely adopted in quantitative analysis because the volume of water required to remove the dialysate is so large that the recovery of the latter in a quantitative manner has been practically impossible. Nevertheless, in the examination of physiological substances, such as blood, milk, animal organs, and secreta, as well as food-stuffs and the like, dialysis may play an important part in the separation of mineral salts, sugars, crystallisable bases, etc., from the colloidal matter in which they may be contained. In designing an apparatus for this purpose (see Fig.), the author has adopted the principle of the well-known Soxhlet extraction apparatus. The water is placed in a boiling-flask, and is continuously distilled up into the top of the condenser, from which it passes down through the dialysing chamber, carrying the dialysed substance into the flask, in which it accumulates. Sufficient water is charged into the apparatus to keep the dialysing cylinder full and the boiling-flask about half-full. In order to have as large a dialysing surface as possible, the dialysing bag is made in the form of an annular cylinder, so that water may pass down one side and up the other on its way to the overflow leading to the flask. In physiological and forensic analyses, using parchment-paper bags, the author was able to recover 98 to 99 per cent. of the dialysable matters in five to seven hours. The heat from the boiling-flask is sufficient to keep the water in the dialysing cylinder warm, thus accelerating the dialysis. For many purposes it is not desirable to subject the dialysed matter to a temperature of 100° C. for such prolonged periods, and in such cases it is advisable to connect the apparatus with a water vacuum pump, by which means distillation is carried on at a temperature of 40° to 45° C. In order to prevent bumping, a slight current of air, regulated by a clip, is admitted through a capillary tube extending to the bottom of the distillation flask.

The apparatus may be obtained from the Vereinigte Fabriken für Laboratoriumsbedarf, Berlin. J. F. B.

Extraction with Liquefied Gases. F. Friedrichs. (*J. Amer. Chem. Soc.*, 1913, **35**, 244-247.)—The apparatus here shown has been devised for extracting substances with liquefied gases. It consists of an extraction vessel, *D*, in which is suspended from three glass projections a container, *C*, and of a condenser, *B*, which is ground to fit into *D*. This condenser is of the screw type, with a hollow, into which the refrigerating material is introduced. Both condenser and extraction vessel are insulated by means of a vacuum jacket, and between this jacket and the inner vessel a small quantity of the refrigerant may be placed. The upper part of the condenser is connected with a gas-cylinder or generator, and with a mercury manometer to indicate the pressure and to act as a safety-valve. For the extraction of substances with liquid ammonia, sulphur dioxide, hydrogen sulphide, methylamine, and similar liquefied gases, solid carbon dioxide and ether may be used as the refrigerant; whilst for gases of lower boiling-point, such as oxygen or methane, liquid air is a suitable refrigerant. In such cases it is advisable to introduce a little ether before the liquid air to prevent risk of breakage. Still lower temperatures may be obtained by connecting the condenser with an air-pump and evaporating the refrigerant under reduced pressure. By the use of this apparatus it was proved that hydrazine sulphate is quantitatively decomposed by liquid ammonia into free hydrazine and ammonium sulphate. A weighed quantity of the pure salt was placed, together with a little glass wool, in a Schleicher and Schüll extraction thimble in the vessel *C*, the thimble being supported on a perforated porcelain plate, and prevented from touching the walls of *C* by means of short pieces of glass tubing. Air was expelled from the apparatus by a current of ammonia, and the refrigerant then introduced into the condenser screw and the space between the vacuum jacket and the apparatus. The tube between the stopcock *G* and the bulb *F* had previously been filled with mercury, and this conveyed sufficient heat to evaporate the liquid ammonia in the bulb *F*. As soon as this bulb had become filled to about three-fourths of its capacity with liquid, the valve of the ammonia cylinder was closed and the extraction then continued automatically. All that was required was to replenish the refrigerant in the condenser screw from time to time. When the extraction was complete the apparatus was allowed to become warm and the gas to escape through the safety outlet. The extract was then drawn off through the stopcock *G*. It is suggested that with an apparatus made of iron instead of glass a large amount of anhydrous hydrazine might be prepared by this method. C. A. M.





Absorption Apparatus for Use in Nitrogen Estimations by Dumas' Method. F. Friedrichs. (*Zeitsch. angew. Chem.*, 1913, **26**, 143.)—The mixture of carbon dioxide and nitrogen obtained in Dumas' method for the estimation of nitrogen in organic substances is collected in the lower part, *A*, of the apparatus shown in the illustration. This part of the apparatus is filled with concentrated potassium hydroxide solution, and is connected with a reservoir; a quantity of mercury, just sufficient to cover the gas inlet, is placed in the apparatus. When the combustion is completed, the tap *C* is turned, so that the nitrogen which has collected in the bulb of the absorption part of the apparatus may be driven upwards into the burette *B*. This is surrounded by a water-jacket, and is filled with water, and the volume of the gas is read off in the usual way. The advantage gained in using the apparatus lies in the fact that the gas is saturated with water-vapour and is measured over water; in the ordinary apparatus the gas is measured over an alkali solution whose vapour pressure varies with its concentration. W. P. S.

New Separating Funnel. C. E. Parker. (*J. Amer. Chem. Soc.*, 1913, **35**, 295-297.)—In order to avoid the formation of emulsions in shaking liquids with immiscible solvents, a form of funnel has been devised in which the surfaces of the liquid in contact are eight to ten times as large as in the funnels of the usual form. With this end in view, the funnel has been made in the form of a shallow plano-convex lens of wide superficial diameter (up to 17·5 cm.). During the extraction the funnel is placed on its flat side upon a disc, which is made to revolve in a nearly horizontal position at the rate of about twenty revolutions per minute, by means of an electromotor. The inclination of the apparatus may be adjusted by means of a screw at a suitable angle to promote a gentle movement of the liquids upon each other. C. A. M.

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REVIEWS.

A HANDBOOK OF SUGAR ANALYSIS: A PRACTICAL AND DESCRIPTIVE TREATISE FOR USE IN RESEARCH, TECHNICAL, AND CONTROL LABORATORIES. By C. A. BROWNE, Ph.D. New York: John Wiley and Sons. London: Chapman and Hall. 1912. Price 25s. 6d. net.

It is not infrequently alleged that methods which are in use for the estimation of sugars and their allied compounds are largely empirical; and whilst this cannot be denied, it must be admitted that these methods have now been brought to such a high state of perfection that the accuracy and precision of which they are capable fall little short of what can be attained with methods applied to inorganic substances which are based entirely on chemical principles. Nor can the reliability of sugar estimations be doubted, seeing that practically identical values can be obtained by means of methods differing so widely in principle as those based on the rotation of the plane of polarised light and on the reduction of cupric salts respectively. The statement, however, needs some qualification in the case of the analysis of the residual products of the sugar factory, where the results are affected by the presence of the impurities derived from the raw juice, as well as by certain of the substances used in the defecation of the latter (both cane and beet products are here referred to), and also because they have been intermittently boiled with these, giving rise to certain products of change. The precise nature of most of these secondary matters is at present unknown to science; consequently the analysis of such residuals can only be interpreted in terms of known sugars, and the results as returned are not so reliable as those obtained with purer products.

Modern developments of sugar analysis are for the most part known only to specialists, and the bulk of those that are published are inaccessible to chemists outside the sugar and fermentation industries. Yet methods of sugar analysis play an increasing rôle in the work of the general analyst, and on this account the want has long been felt for a practical handbook describing the most approved methods in this branch of technology. The author of the present volume is eminently qualified to write such a treatise. Originally a student of Professor Bernard Tollens of Göttingen (who has added so much to our knowledge of the chemistry of the sugars and allied compounds), Dr. Browne has acted successively as chief of the sugar laboratory of the U.S. Bureau of Chemistry, Washington, and as research chemist of the Louisiana Experiment Station, New Orleans, whilst at the present time he occupies the position of chemist to the New York Trade Laboratory. The work is indeed to be regarded as a valuable addition to the literature. Covering 787 pages, together with 100 pages of tabular matter, it cannot be denied that the text is written for the most part clearly and concisely. It is to be regretted, however, that some branches of the subject receive either no notice at all or are dealt with in a very perfunctory manner, whilst some 260 pages (forming the second part of the volume) are devoted to matters of pure chemistry which are altogether outside the scope covered by the title. With this part of the work it is not proposed to deal in detail.

Some excellent directions are given for sampling sugars and sugar products, and attention is called to the changes in composition which occur owing to the loss or gain of water, as well as to the action of enzymes and micro-organisms, considerations the importance of which will be conceded when it is remembered to what extent the so-called "polarisation value" of raw cane sugars (on which the duty is based in this country) is affected during their transit.

The description of physical methods forms, perhaps, one of the most important features of the work, and special mention in this connection must be made of the chapters on the principle and use of the refractometer and of the polarimeter. To some it might appear that a description of the measurement of osmotic pressure by plasmolysis was extraneous, as it has no direct bearing on sugar technology; however, many will no doubt find its inclusion acceptable. The use of the refractometer in determining the percentage of solid matter in sugar products has, when expedition is essential, advantages over the ordinary methods. Refractometric methods are also superior to densimetric methods, as their accuracy is affected to a lesser extent and by a smaller number of the extraneous substances present in commercial sugar products. As the author very truly remarks, H. Main was the first to demonstrate the practical utility of the Abbe refractometer in sugar-house work as an accurate measure of the moisture and total solids in all products except the very lowest. It may here be pointed out, moreover, that the more recent determinations of Schönrock ("Physikalisch-Technische Reichanstalt," Berlin) accord remarkably with those of Main. It is to be regretted that the author occasionally refers to quartz-compensating saccharimeters as "polariscopes," since they have an equal right to the more accurate term "polarimeter," as those instruments in which measurements are made by the direct rotation of the analyser. It is well known that in polarimetric methods of estimating sucrose in raw sugars depending on both single and double observations, the use of clarifying agents introduces numerous errors, and much work has been conducted during recent years towards the elimination of these errors. Dr. Browne must be congratulated on the full and accurate account which he has given to this part of the subject. Under physical methods some mention should, in the writer's opinion, have been made of the method of estimating ash in sugar products by determining their electrical conductivity, which was first elaborated by Main, and more recently improved by Lange.

The analysis of sugar beets is quite adequately dealt with, and most of the Continental methods are described. With regard, however, to the pulping of the roots for analysis, Keil's boring rasp only is illustrated, whilst no mention is made of Kiehle's rasping machine, which is designed to take out a longitudinal section right through the middle of the root; and in the writer's opinion this is the most satisfactory of any machine on the market, it being used exclusively in his laboratories.

Of cupric reduction methods for the estimation of the sugars, all the leading gravimetric methods are given, whilst volumetric methods are dealt with very sparsely. Seeing that so much of the so-called "dextrin-maltose" or "glucose syrup" is manufactured in America, it is not a little surprising to find no mention of this product in the book, especially in view of its wide use in confectionery. The writer has frequently to estimate the quantity of glucose syrup present in admixture with other sugars, and he has always found fermentation to give satisfactory results.

Dr. Browne, however, in mentioning fermentation as a means of eliminating sugars from fruit products containing dextrin, gives as an objection to the method that most yeasts ferment or modify dextrin to a greater or less degree. For the analysis of starch conversion products, a method originally described in a former edition of Allen's "Commercial Organic Analysis" is reproduced. Dr. Browne, it is true, throws some doubt on the accuracy of this method, but as an expert on the subject he ought to know that it could not give results with any approach to accuracy. The method recommended for the estimation of so-called "dextrin" in honey is one of precipitation with alcohol. Honey dextrin is said to have a specific rotatory power of $[\alpha]_D + 115°\text{-}160°$.

Methods have been devised for the detection of cane-sugar in maple products depending on the measurement of the basic lead acetate precipitate, and the author, after devoting more than two pages to their description, quotes an experiment of Doolittle and Seeker exposing their fallacy. The writer cannot agree with Dr. Browne's statement that one of the most valuable means of ascertaining the source of a sugar is to determine the composition of its ash. This is a long-since exploded notion. It is, however, quite true that beet products are more apt than cane to contain nitrates.

Many parts of this book leave nothing to be desired, and it is to be hoped that Dr. Browne at no distant date will remodel the whole, leaving out many portions of the text on which information can be found in other treatises, and filling in certain lacunæ. This will render his book even more valuable than it is at present.

ARTHUR R. LING.

DIGITALIS ASSAY. By W. H. MARTINDALE, Ph.D. H. K. Lewis. London. 1913. Pp. 47. Price 2s. net.

In this communication to the Pharmaceutical Society, now published in book form, a comparison is made between the physiological and chemical methods for the assay of digitalis, and a new and simple method is put forward for the approximate chemical assay.

The first eleven pages are devoted to a brief review of the present knowledge of the digitalis glucosides and of the methods suggested for their isolation or determination, and subsequently some seven pages are occupied with the chief methods of physiological standardisation which have been suggested or are in actual use.

When it is realised that the therapeutic effects of digitalis are due to at least four glucosides, about which not very much is known; that these are not uniformly distributed throughout the plant, and differ as regards both the nature and the power of their action; and, further, that the proportion in which they occur, and consequently the activity of the preparations, varies with the age of the plant, the soil and climatic conditions under which it is grown, and the time at which it is gathered, it will be readily admitted that a satisfactory process for their assay is as greatly needed as it is difficult to devise.

The chemical processes hitherto employed for the standardisation of the preparations have really been nothing more than rough colorimetric determinations of the digitoxin. The Pharmacopœias of Great Britain, the United States, Belgium, and Denmark contain no directions for the assay of the drug or its preparations, and the suggestions put forward in the other Pharmacopœias have reference mainly to the Keller-Kiliani test, which is a colour test for digitoxin, and takes little or no account of the water-soluble glucosides, on which so much of the activity of digitalis depends.

The physiological tests which are at present mainly used for standardising the tincture cannot be regarded as much more satisfactory, inasmuch as they really afford a standard of toxicity rather than of therapeutic value. The fact that the results of physiological trials by no means always correspond with those of chemical examination, though not necessarily an objection, is certainly not a reassuring factor.

Apart from the uncertainty of any method based on experiments on animals so low in the scale as frogs, it is obviously desirable that some simpler plan, not requiring the aid of an expert physiologist, should be available. A method which really only gives the information that a certain volume of the tincture under examination will kill 100 grms. of "frog" in, say, twelve hours does not inspire too much confidence when it is realised that the result varies with the species of frog, its age, its size, its sex, and its condition. Nevertheless, it has proved of great value in the past, and is the best at our disposal at the present time.

The author's new test aims at making the pharmacist independent of the physiologist, and is a great advance on the Keller-Kiliani process, as it takes into account the curative constituents on which the action of the drug mainly depends—*i.e.*, the water-soluble glucosides.

After a preliminary treatment to remove various interfering substances, 0·1 c.c. of the product is mixed with Frohde's reagent, and should then produce a pink colour varying in intensity with the proportion of soluble glucosides present. By comparison with a colour scale, reproduced in the text, the strength in terms of the physiological test is ascertained. A further 0·1 c.c. of the extract is mixed with glacial acetic acid and poured on the surface of the Frohde's reagent, when a blue ring, indicative of digitoxin, should be obtained.

As the result of numerous experiments the author states that "it was possible to obtain depths of colour comparing most favourably with the physiological data, and it is claimed that the method can be made to yield results which may obviate physiological assay of digitalis tincture by means of frogs."

From the point of view of the analyst the method is obviously open to criticism. There is always an element of uncertainty about colorimetric processes, and when comparison has to be made between a liquid in a test-tube viewed partially at least by transmitted light, and a patch of colour on paper viewed entirely by reflected light, this uncertainty is not decreased. The fact, moreover, that so much depends on the accurate measurement of such a small volume of liquid as 0·1 c.c. is a further drawback.

Nevertheless, the work is a step in the right direction in so far as it attempts to measure the really curative constituents of the drug, and in expert hands the process may prove on further experience to be all that the author anticipates.

The monograph in any case embodies the results of much labour; it deals with many points of interest in connection with digitalis, apart from the question of standardisation, and is a valuable contribution to the literature of a very complicated subject. C. H. CRIBB.

CHEMISTRY FOR DENTAL STUDENTS. By H. CARLTON SMITH. New York: John Wiley and Sons. London: Chapman and Hall, Ltd. 1912. Second edition. Price 10s. 6d. net.

This book is divided into eight parts, dealing with qualitative analysis, dental metallurgy, volumetric analysis, microchemical analysis, organic chemistry, physiological chemistry, digestion, and urine, and "follows closely the lecture course in dental chemistry at the Harvard Dental School." "Its aim has been to give the student, *as concisely as possible*, such portions of the various branches of chemistry as are most likely to be of value in practical work." We doubt very much if this aim has been achieved; it would rather appear that, in attempting to cover so much ground and to be concise, the author has only succeeded in producing a book which, though possibly of use to a student in preparing for certain set examinations, would be certain to produce a bad form of chemical indigestion. As an example of the sacrifices made on behalf of conciseness, we may mention that malonic acid is dismissed in two lines, with the observations that it "is an oxidation product of malic acid (from apples), and is comparatively unimportant."

The student using the book is supposed to have had a laboratory training in general chemistry, and to be conversant with the principles of theoretical and physical chemistry, yet it is considered necessary in an introductory chapter to revise some definitions, such as element, compound, atom, molecule, etc., and here we find that in the symbol "H—O—H the *dash* (*bond*) shows that oxygen has two combining points relative to the hydrogen, which is considered to have one"; and in the section devoted to qualitative analysis the following are among many similar instructions:

"Break point of filter-paper with glass rod and wash Group 2 into beaker with warm $(NH_4)_2S$."

"Filter and wash. Throw away wash-water. Pierce filter and wash sulphide into large test-tube or small beaker."

Is such information really necessary for students who have had a laboratory training in general chemistry?

The book does not appeal to the reviewer, and he cannot recommend it to students desirous of obtaining an adequate training in the principles of chemistry as applied to dental science. ARTHUR W. CROSSLEY.

EAUX-DE-VIE: EAUX-DE-VIE NATURELLES ET INDUSTRIELLES. By X. ROCQUES. Pp. 316 + Table of Contents. Paris: Ch. Béranger. 1912. Price 6 francs.

This little volume is one of a series of "Manuels Pratiques d'Analyses Chimiques," edited by MM. Bordas and Roux. Chapter i. deals with the nature of various spirits, chapter ii. with their analysis, and chapter iii. consists of a series of tables of analytical results interlarded with critical notes. The work concludes with a number of appendices, dealing (1) with spirit legislation in various countries, and (2) with official methods of analysis. Chapter i. contains, in tabular form, an interesting series of suggestions regarding the denomination of various types of blended spirits, but it does not appear that this classification is official. The section dealing with analytical methods is brief, covering some thirty-six pages, but is no doubt suited to the requirements of French official practice. The collection of tables in chapter iii. should be useful to those interested in the constitution and analysis of French spirits, but the information regarding other spirits is meagre. The appendices give the reader a very fair idea of legislation in a number of countries, but it is not quite clear why the résumé of official methods of analysis should have been restricted to data concerning Belgium and Italy.

P. SCHIDROWITZ.

INTRODUCTION TO THE RARER ELEMENTS. By P. E. BROWNING. Third edition. Pp. xii + 232. New York: John Wiley and Sons. London: Chapman and Hall. 1912. Price $1.50.

The third edition of this useful little book is an improvement in several respects on the earlier editions. The chapter on qualitative separation has been considerably enlarged, and may be commended as an admirable summary of work which in the original memoirs is too voluminous to be directly useful, and much of which is of very recent date. The chapter on the technical application of the rarer elements has also been expanded, and a table of spectroscopic lines, and plates showing typical spectra, have been added. The index is deserving of imitation. Arranged in tabular form, it occupies only two pages, but contains as much information as an ordinary index of seven times that length, and contains it in a more readily accessible form.

The analyst must not expect to find in this small volume detailed directions for the quantitative separation and estimation of the rarer elements. For example, he would look in vain for a method for the estimation of vanadium in presence of chromium, a commonly occurring problem, formerly very difficult of accomplishment, but to-day comparatively easy. Similar instances might be multiplied, but this would not be fair to the author, who described his book, in the preface to the first edition, as a "handbook in the introductory study of the rarer elements." The book is addressed to senior students, not to analysts, but as a first reference, where one of the really rare elements is concerned, it may well prove useful to the analyst. Its utility would be enhanced by more frequent reference to original memoirs in which difficult quantitative separations are dealt with in detail, and the existing references would be more handy if the dates as well as the volume numbers of periodicals were given.

G. C. JONES.

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INSTITUTE OF CHEMISTRY.

PASS LIST: MARCH—APRIL (1913) EXAMINATIONS.

OF twenty-five candidates who presented themselves for the final examination fourteen passed. In the Branch of Mineral Chemistry: S. C. Bate, B.Sc. (Lond.), F. G. Crosse, and S. G. Greene; in the Branch of Metallurgical Chemistry: E. Marsden; in the Branch of Organic Chemistry: W. F. Hollely, T. S. Jones, B.Sc. (Lond.), H. Lambourne, B.Sc. (Lond.), D. H. Peacock, B.Sc. (Lond.), B.A. (Cantab.), and F. Sproxton, B.Sc. (Lond.); in the Branch of the Chemistry of Food and Drugs, and of Water: A. L. Davidson, R. E. Griffiths, B.Sc. (Lond.), F. W. Hoyland, W. G. Saunders, and W. A. Storey.

JUNE, 1913. Vol. XXXVIII., No. 447.

THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

THE monthly ordinary meeting of the Society was held on Wednesday evening, May 7, in the Chemical Society's Rooms, Burlington House. The President, Mr. L. Archbutt, F.I.C., occupied the chair.

The minutes of the previous ordinary meeting were read and confirmed.

A certificate of proposal for election to membership in favour of Mr. F. S. Sinnatt was read for the second time; and certificates in favour of Messrs. Charles William Birch, 550, Simcoe Street, Victoria, B.C., Public Analyst for the City of Victoria, British Columbia; Ulick Richardson Evans, B.A. (Cantab.), 28, Victoria Street, Westminster, Consulting Electro-Chemist; and Harold Warren Gill, B.Sc. (Lond.), A.I.C., South African School of Mines, Johannesburg, were read for the first time.

Messrs. H. F. E. Hulton, A.I.C., C. T. Kingzett, F.I.C., and A. J. Parker, were elected members of the Society.

The following papers were read: "A New Apparatus for Maintaining Constant Temperatures," by F. H. Dupré and P. V. Dupré, F.I.C.; "On the Proportionate Determination of Cocoanut Oil and Palm-Kernel Oil in Mixtures," by H. R. Burnett and Cecil Revis; "Examination of the Oils from *Manihot ceara* and *Funtumia elastica*, and a Comparison of their Properties with those of Linseed and Hevea Oils," by S. Rideal, D.Sc., F.I.C., and L. H. D. Acland, B.A.; "The Recovery of Iodine from Residues," by Harold W. Gill, B.Sc.; and "The Composition of Milk," by H. Droop Richmond, F.I.C.

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OBITUARY.

WE greatly regret to record the death, on April 29, of Dr. M. A. Adams, of Maidstone, a past President of the Society. An obituary notice will appear in the next issue of the Journal.

EGYPTIAN BUTTER AND SAMNA.

By S. H. TRIMEN.

(*Read at the Meeting, April* 2, 1913.)

In Egypt, as in all Eastern countries, butter-fat is used by the native population, and not butter as it is known in Europe. This butter-fat, called "ghee" in India, is known in Egypt under its Arabic name "samna" (or more correctly "samn"). In the course of the following paper the commercial product will be referred to under that name. Samna is usually made in the following way: The whole milk is churned by shaking it up in goatskins until the butter (Arabic, "zibda") forms. This part of the process is done by the fellahin in their villages; but because it is not economical to make samna in small quantities, they do not make it themselves, but sell the zibda to the samna makers. These latter usually live in the towns, and send their agents round the neighbouring villages to buy the zibda from the fellahin. The zibda which has been collected in this way is heated in large pans until it melts; the water, casein, etc., subside, the samna is poured off and usually stored in old kerosene tins.

The chief source of Egyptian samna is the milk of the buffalo ("gamoosa"). Cow's milk is probably used only to a limited extent, the number of cows in Egypt at the present time being comparatively small, and their milk being largely used for drinking purposes. Although there are large numbers of sheep and goats in Egypt, their milk is used almost exclusively for making cheese, and is not used to any extent in the manufacture of samna. It is quite likely that from time to time small quantities of zibda from cow's, sheep's and goat's milk go to the samna makers, but the amount is apparently not large enough appreciably to affect the analytical constants of normal Egyptian samna, although it may account for an occasional abnormal sample. Syrian samna, which is largely imported into Egypt (1,073,694 kgrms. in 1910 and 676,051 kgrms. in 1911), is, on the other hand, generally believed to be made from sheep's and goat's milk. Its actual composition will be discussed later, but it is unlikely that cow or buffalo milk is used in any quantity in its manufacture. Indian ghee, which is also imported into Egypt, is apparently made from buffalo's milk, though cow's milk may enter occasionally into its composition. Samna is only made on a large scale during the winter months, and it is the custom amongst the more wealthy of the Egyptians to purchase their annual supply at that season and store it in a cool place during the year. Being for all practical purposes a pure fat, its keeping properties are, of course, vastly superior to those of butter; indeed, it is not uncommon for samna to be used as a food some years after its manufacture, but reference to this will be made later on.

Samna as it is known in Egypt is pale yellow in colour, possessing a smell, sometimes cheesy, sometimes acid, frequently rancid, and always distinctly unpleasant to European ideas. Indian ghee is very similar, but Syrian samna frequently has a not unpleasant smell suggestive of ethyl butyrate; the writer, however, has not been able to discover whether this smell is natural to the samna or due to the addition of a small quantity of an ester. During the cold weather samna is a solid opaque grease with a granular consistency; in summer it is sometimes liquid, but is usually in a semi-liquid state. Butter proper, as distinct from samna, is made in Egypt all the year round, but only in the large towns, for the use of European residents. As buffalo butter is very pale in colour, it is the usual practice for the dairyman to make it resemble cow butter by the addition of a small quantity of butter colouring. Apart from such sophistication which may, or may not, be justifiable, the adulteration of butter and samna is common in Egypt. Cocoanut oil and clarified animal fat are the usual adulterants, and enormous quantities are imported into Egypt for this purpose—1,397,139 kgrms. of cocoanut oil were imported in 1911—in fact, a large recognised trade exists between the importer and the samna and butter maker, fostered by the former's assiduous and plausible agents. Both sesame and cottonseed oil have been detected occasionally in adulterated samna; it is not known, however, whether they are added directly to samna, or whether they enter it as ingredients of imported butter adulterants, possibly "earmarked" by their means before exportation. A few examples of adulterated samna are given in Table I., and these samples, which are typical, show the large proportion of adulterant usually added by the Egyptian sophisticator.

Since the examination of butter and samna in Egypt presents many features not encountered in normal analytical practice, it was not at first easy to decide which of the constants it would be most advantageous to determine in routine work. At the present time the writer determines the Reichert-Meissl, Polenske, saponification and Valenta values, the Zeiss butyro-refractometer reading at 40° C., and tests qualitatively for sesame and cottonseed oil. As the ideal method of testing butter is still to be found, this list is in no way final. Other and newer methods have been tried in the hope of finding a routine test more particularly suitable to the local conditions, but without much success. An account of the trial of these methods with samna is now given:

The Avé Lallement (Baryta) Method.—This method was tried on some forty samples of samna, butter, possible adulterants, and mixtures containing adulterants. Some of the results, together with the other analytical constants determined, will be found in Table I.

In every case genuine Egyptian butter and samna were found to give negative values for $b-(200+c)$, thus showing that Lallement's statements apply to butter-fat from the buffalo as well as that from the cow. Unfortunately, however, these values are in many cases so low that a negative value is still given after the addition of a considerable amount of cocoanut oil. It has been found that the addition of 10 per cent. cocoanut oil to a samna increases the value of $b-(200+c)$ on an average by 6·2. This means that if the $b-(200+c)$ value of the samna is less than $-6{\cdot}2$, the samna will still give a negative figure for this value after the addition of 10 per cent. cocoanut oil, and it is obvious that when the $b-(200+c)$ value is as low as -20, as it sometimes is, as much as 30 per cent. cocoanut oil could be added without its detection by this method. It is also stated by Revis and Bolton (Allen's "Commercial Organic Analysis," fourth edition, vol. ii., p. 289) that in cases where negative values are obtained for $b-(200+c)$ with mixtures of cocoanut oil and

TABLE I.

No.	Description.	Reichert-Meissl.	Polenske.	Zeiss at 40° C.	Valenta (Acetic Acid: sp. gr. 1·060.)	Saponification Value.	Baryta Value.				Sesame Oil.	Cottonseed Oil.
							a.	b.	c.	b − (200 + C.)		
1	Genuine Egyptian samna ...	31·9	2·2	41·7	—	231·3	316·2	256·0	60·2	− 4·2		
2	" " " ...	34·1	1·5	42·1	—	230·0	314·5	253·1	61·4	− 8·3		
3	" " " ...	33·4	1·7	42·2	—	230·9	315·6	255·3	60·3	− 5·0		
4	" " " ...	33·3	2·1	42·0	—	229·3	313·5	253·0	60·5	− 7·5		
5	" " " ...	33·0	2·0	41·7	—	228·3	312·1	252·9	59·2	− 6·3		
6	" " " ...	32·1	1·4	42·0	—	228·1	311·8	253·8	58·0	− 4·2		
7	" " " ...	31·6	1·9	42·0	58	230·9	315·8	248·9	65·4	− 16·5		
8	No. 7 + 10 per cent. cocoanut oil	29·6	2·8	41·3	55	232·6	317·9	252·9	65·0	− 12·1		
9	No. 7 + 20 per cent. "	27·4	4·1	40·6	50	235·6	322·1	259·1	63·0	− 3·9		
10	No. 7 + 30 per cent. "	24·9	5·0	39·8	47	237·3	324·4	265·3	59·1	+ 6·2		
11	Genuine Egyptian samna ...	36·6	2·3	41·7	—	236·3	323·0	251·5	71·5	− 20·0		
12	No. 11 + 10 per cent. cocoanut oil	33·7	3·1	40·6	—	237·4	324·6	256·2	68·4	− 12·2		
13	Genuine Syrian samna	24·4	4·1	42·9	65	227·3	310·7	254·7	56·0	− 1·3		
14	" " "	27·2	5·3	43·0	60	232·8	318·2	254·6	63·6	− 9·0		
15	" " "	31·2	6·4	42·3	—	235·5	322·0	253·9	68·1	− 14·2		
16*	Goat butter-fat	20·8	6·5	40·7	—	231·6	316·6	267·1	49·5	+ 17·6		
17*	" "	22·9	4·9	43·6	—	224·5	306·9	253·5	53·4	+ 0·1		
18*	Sheep butter-fat	22·9	2·8	44·0	75	223·2	305·1	256·4	48·7	+ 7·7		
19*	" "	26·5	3·1	44·0	70	216·6	296·1	253·0	43·1	+ 9·7		
20*	Buffalo butter-fat	32·3	1·6	42·0	—	228·4	312·2	251·8	60·4	− 8·6		
21*	Butter-fat from milk of one cow	23·4	1·6	46·5	—	217·1	296·8	251·8	45·0	+ 6·8		
	ADULTERATED SAMNA.											
22	Bought in Cairo	10·4	8·5	39·9	—	233·5	319·2	282·9	36·3	+ 46·6	−	−
23	" "	18·2	7·3	40·0	—	236·3	323·0	275·6	47·4	+ 28·2	+	−
24	" "	12·8	10·3	39·0	—	238·5	326·0	282·3	43·8	+ 38·5	+	−
25	Bought in Alexandria	5·0	4·3	43·7	—	216·6	296·1	276·1	20·0	+ 56·1	+	trace
	BUTTER ADULTERANTS.											
26	Cocoanut oil	7·2	16·9	35·0	—	254·7	348·2	296·9	51·3	+ 45·6	−	−
27	Mutton-fat	—	0·3	47·2	—	194·5	265·8	264·0	1·8	+ 62·2	−	trace
28	Lard	—	0·3	50·0	—	193·1	264·0	263·7	0·3	+ 63·4	−	+
29	Cottonseed oil	—	—	58·8	—	185·3	253·3	235·1	18·2	+ 16·9	−	+
30	Sesame oil	—	—	61·6	—	193·8	264·9	247·1	17·8	+ 29·3	+	−

* Prepared in laboratory.

butter-fat that the value of *b* for these mixtures always exceeds 260. From theoretical considerations alone, it is difficult to see how this can always be the case. In genuine butter-fat *b* is frequently below 250, and according to Avé Lallement 10 per cent. cocoanut oil only increases the value of *b* by from 4·5 to 5. Hence if 10 per cent. cocoanut oil be added to a butter-fat, with, say, a *b* value of 250, the value of *b* can only be increased to a maximum of 255—*i.e.*, considerably below 260. It is also curious to find on the very page on which this statement is made an example given of a butter containing 10 per cent. cocoanut oil with *b* value of 259·2. However these discrepancies may be explained, the statement that mixtures of cocoanut oil and butter-fat always give values for *b*, exceeding 260, does not hold good for samna. In Table I. will be found two samples containing 10 per cent. cocoanut oil (Nos. 8 and 12) and one containing 20 per cent. (No. 9), all of which give lower *b* values than 260. In his original paper Avé Lallement states that the method breaks down with overheated or rancid butter-fat. It is possible, therefore, that the failure of the method in these cases may be due to the samna having been overheated in its manufacture. Whether this be so or not, it is difficult to understand how any butter-fat with a maximum figure of $-23·8$ for the $b-(200+c)$ value, such as is given by Avé Lallement, can ever give a positive reading for that value after the addition of a small quantity of cocoanut oil.

Another fact which detracts from the value of this method in an Eastern country is that both goat and sheep butter-fat give at times positive values for $b-(200+c)$. The figures (Table I., Nos. 16 to 19) were obtained from butter made in the laboratory from mixed genuine milk of sheep and goats. Two samples of each were examined. The two samples of goat butter-fat gave $+17·6$ and $+0·1$ for the $b-(200+c)$ value, and the two samples of sheep butter-fat $+7·7$ and 9·7 respectively. It is not claimed that these results are in any way typical; they are simply to show that the method does not always hold good with butter-fat other than that of the cow and buffalo.

Curiously enough, the only samples of Syrian samna—believed to be made chiefly from sheep milk—examined by this method all gave negative values for $b-(200+c)$, viz., $-1·3$, $-9·0$, and $-14·2$ (Table I., Nos. 13 to 15).

In view of the many shortcomings of this method, which have been sufficiently indicated above, the writer cannot endorse the high opinion of its value which has been expressed by Revis and Bolton (ANALYST, 1911, **36**, 393). The writer is of the opinion that the information given by it is not sufficient to justify its adoption as a routine method for the testing of butter and samna in Egypt, but that as a confirmatory test in suspicious cases it might occasionally be of value.

The Shrewsbury-Knapp Method.—In using this method the writer, in common with other workers (ANALYST, 1911, **36**, 334, and 1912, **37**, 6) experienced great difficulty in obtaining concordant results. This difficulty was doubtless in part due to the variable temperature experienced. Indeed, it seems improbable that a method which depends on the solution of a substance at a definite temperature can ever be employed as a routine method in a country such as Egypt, where it is no uncommon occurrence for a room temperature to vary 25° F. in the course of a morning.

The modification suggested by Revis and Bolton (ANALYST, 1911, **36**, 334) has not been tried up to the present.

The Fendler Alcohol Solubility Value (ANALYST, 1910, **35**, 355).—The writer i not at present in a position to give a definite opinion on this method, although several determinations have been made. In common with the Shrewsbury-Knapp method, its chief defect is that it depends on obtaining a solution at a constant temperature, which naturally militates against its use in a hot climate. It is hoped that the introduction of certain modifications to remove the temperature difficulty will render it a valuable confirmatory test. Its great advantage is that, suitably modified, it is very little trouble to carry out, being merely a continuation of the Reichert method, no additional quantity of butter-fat having to be weighed out.

Colour Test for Cocoanut Oil.—Ludwig and Haupt's qualitative test (*Zeitsch. Untersuch. Nahr. Genussm.*, 1907, **13**, 605-610) was tried, but unfortunately with negative results.

The chief object in trying the above methods was to find one that would enable the analyst to distinguish readily between different varieties of butter-fat. To show how extremely useful it would be to have such a method, it is only necessary to point out that the analytical constants of sheep butter-fat (*vide* Syrian samna) resemble very closely those that would be given by a mixture of cow or buffalo butter-fat with cocoanut oil. So far, unfortunately, no quantitative method has been found by which different kinds of butter-fat can be detected. Very useful indications, however, have been given by Hinks' test (ANALYST, 1907, **32**, 160), a qualitative method originally devised for the detection of cocoanut oil in butter. Different varieties of butter-fat and mixtures of butter-fat with cocoanut oil were examined by this method, and it was found that the final products, when viewed under the microscope, differ with different kinds of butter-fat. The following is a description of the microscopical appearance of the various samples examined:

Buffalo Butter-fat (Egyptian Samna). — Small yellow globules, more or less rounded and homogeneous, but with no crystalline structure.

Cocoanut Oil.—Fine white needles, growing concentrically, markedly resembling the conventional geographical map-sign for mountains.

Buffalo Butter-fat + 10 *per cent. Cocoanut Oil.*—Small yellow globules, most of which are surrounded with small hair-like crystals, which appear to be growing out of the globules. Their appearance resembles that of a ciliated unicellular organism.

Buffalo Butter-fat + 50 *per cent. Cocoanut Oil.*—The appearance approximates very closely to that of pure cocoanut oil, the difference being that the crystals are yellowish not white; there is also a certain amount of amorphous material besides the crystals.

Goat Butter-fat.—A very similar crystalline growth to that from cocoanut oil, only that the crystals are smaller and slightly yellowish. It strongly resembles a butter-fat containing a large percentage of cocoanut oil.

Sheep Butter-fat.—Globules, larger, lighter in colour, and more homogeneous than is the case with buffalo butter-fat. They have no crystalline structure, and are frequently split across in a manner resembling some of the larger varieties of starch granules.

Syrian Samna.—The two samples of Syrian butter-fat examined by this method both gave results identical with that obtained from sheep-butter, no trace of crystalline structure being observed.

These results appear to justify the following conclusions :

1. In the absence of cocoanut oil, it is possible by this method to distinguish between buffalo or cow butter-fat, and that from sheep and goats.

2. Owing to the close resemblance that exists between the product from cocoanut oil and goat butter-fat, it is impossible to detect the presence of cocoanut oil in goat butter-fat or the presence of goat butter-fat in cocoanut oil. Either being absent, the test is of considerable value as a confirmatory test for the other.

3. Some Syrian samna is apparently made entirely from sheep milk. If this were always the case, and if goat's milk were never a source of Syrian samna, then its adulteration by means of cocoanut oil could be confirmed by this method. Unfortunately, it is generally believed that goat as well as sheep butter-fat enters largely into the composition of Syrian samna, and until a large number of samples have been examined this cannot reasonably be disproved.

The Hinks' method, unfortunately, has one great disadvantage, which detracts considerably from its usefulness. Owing to the low melting-points of the globules and crystals, which have to be examined microscopically, it is practically impossible to use the method in hot summer weather. A cooled stage was tried, but was found to be quite impracticable owing to the condensation water which immediately formed on the cover-glass, completely obscuring the object. The test is, therefore, only of value in Egypt during the winter months.

Before considering the analytical constants of butter-fat in Egypt, it is as well to state that the Reichert-Meissl, Polenske, saponification, and Zeiss values, are all strictly comparable with those obtained in English analytical practice. The Valenta values were, on the other hand, obtained by using acetic acid of sp. gr. 1·060, which happened to be the strongest glacial acetic acid in the laboratory when the tests were started. It has been used for all the tests, so that the results, although comparable among themselves, cannot be compared with results obtained by using acid of a different strength.

In Table II. will be found the mean results obtained from Egyptian butters and butter-fats during the past few years, arranged according to their ascending Reichert-Meissl values. All the results here tabulated were obtained from samples believed to be genuine:

The Reichert-Meissl Value, it will be noticed, is much higher than that normally obtained from cow butter, and shows a variation from 28 to 38. These are limiting values, the normal value, which will be discussed later, lying within closer limits. *The Polenske Value* varies directly with the Reichert, the variation being, however, very much less than in the case of cow butter. The minimum value observed by the writer was 1·1, and the maximum 2·4 ; both these values, however, are abnormal. A variation of 10 in the Reichert-Meissl value was found to correspond with an average variation in the Polenske of 0·7, as against 2·0 with cow butter. Such a comparatively small variation considerably enhances the value of the Polenske figure in the detection of adulteration. Taking the mean of a number of experiments, the writer has found that the addition of 10 per cent. cocoanut oil to a butter-fat

TABLE II.

Mean Results from Egyptian Samna and Butter, arranged according to the Reichert-Meissl Values.

Number of Samples.	Reichert-Meissl (Limits).	Polenske.	Zeiss at 40° C.	Valenta (Acetic Acid, Sp. Gr. 1·060).	Saponification Value.
3	28–29	1·3	42·5	70·5	225·0
5	29–30	1·6	42·9	66·0	225·9
7	30–31	1·5	42·1	67·0	227·8
9	31–32	1·8	42·1	62·0	229·5
14	32–33	1·8	42·1	61·0	229·5
13	33–34	1·7	42·0	61·0	229·8
14	34–35	1·8	42·0	59·0	230·9
5	35–36	2·0	42·1	58·0	231·6
6	36–37	2·0	42·0	53·0	233·8
3	37–38	2·0	41·8	53·0	234·9

raises the Polenske value by 1·4—that is, by twice the normal variation of the Polenske value. Hence the detection of small amounts of cocoanut oil in butter and samna of known Egyptian origin becomes a comparatively easy matter. *The Saponification Value* varies directly and very regularly with the Reichert-Meissl figure. Plotted graphically, it gives what is practically a straight-line curve. Such a regular variation is, of course, very useful in forming an estimate of the genuineness of suspicious samples. *The Valenta Value* varies inversely and fairly regularly with the Reichert. As will be seen from the table, the variation is considerable, and were the analyst unaware of it he might frequently be unduly suspicious of abnormal samples. *The Zeiss Butyro-Refractometer:* Except at the extreme limits, where slight variation is shown, this value is practically constant at 42.

TABLE III.

Results showing Seasonal Variation in the Composition of Egyptian Butter.

Month.	No. of Samples.	Reichert-Meissl.	Polenske.	Zeiss at 40° C.	Valenta (Acetic Acid sp. gr. 1·060).	Saponification Value.
August ...	3	30·5	1·3	42·0	67·5	227·3
September	2	30·9	1·3	42·0	64·0	228·5
October ...	—	—	—	—	—	—
November ..	5	33·2	1·4	42·0	64·5	229·9
December ...	4	32·0	1·6	42·5	67·0	228·8
January ...	5	35·0	1·9	42·5	62·0	231·5
February ...	4	35·2	1·9	42·0	58·5	231·6
March ...	4	34·4	1·9	42·0	57·0	231·0
April ...	4	34·0	1·8	42·0	59·0	231·1
May ...	4	33·6	1·7	42·0	60·0	231·1

Seasonal Variation.—In such a country as Egypt it is only to be expected that the composition of butter-fat will vary considerably during the course of the year. That this is the case is shown in Table III. The figures there given are the mean results of weekly samples of butter (not samna) kindly supplied by Mr. Howie of the Cairo Hygienic Dairy. The information it supplies, however, although incomplete, is sufficient to indicate the general tendency of the variation which takes place during the course of the year. It shows (1) that the Reichert-Meissl, Polenske, and saponification values are at their lowest in the summer, and at their highest in the winter; (2) that the Valenta value varies inversely with these values—that is, it is highest in the summer and lowest in the winter; (3) that the Zeiss reading remains practically constant. These changes are to some extent, no doubt, directly due to the effects produced on the animals by the great seasonal variation of temperature, but it seems probable that they are mainly brought about by differences in diet. During the summer months in Egypt there is an almost entire absence of green fodder, the cattle being mainly fed on chopped straw ("tibn"), while in winter there is practically an unlimited supply of green food.

The chief green fodder of the country is a lucerne (*Medicago sativa*), known locally as "berseem," which flourishes from November to May. It is during these months that the cow-buffaloes give their maximum and richest supply of milk, and it is then that most of the year's supply of samna is made. On looking through Table III., it will be seen that during these months the analytical constants show comparatively little variation, and it is therefore reasonable to suppose that normal Egyptian samna, when it is fresh, will show similar slight variations. This is undoubtedly the case, the normal variations being as follows: Reichert-Meissl, 30 to 36; Polenske, 1·4 to 2·0; saponification value, 228 to 232; Valenta, 57 to 64; and Zeiss practically constant at 42.

The Alteration in Composition of Samna on Keeping.—Crispo (*Bull. Soc. Chim. Belg.*, 1910, **24**, 436-438) has pointed out that on keeping butter-fat in closed flasks for a period of one to three years there is usually a slight gain in the proportion of volatile acids (Reichert-Meissl values). Before Crispo's paper appeared, the writer had had occasion to examine certain samples of samna which gave unusually high saponification values and correspondingly low Valenta values, but which were otherwise normal, and apparently genuine. As the samples all had a rancid smell, it was thought that the abnormal figures might possibly be due to the effect of age. It was therefore decided to keep a few samples of fresh samna in glass-stoppered bottles, and to determine after a lapse of time whether any alteration had occurred in their composition. Of the three samples taken, Sample A was kept for two years and examined at the end of each, and Samples B and C were only examined at the end of one year. The results are given in Table IV. They are interesting for several reasons. In the first place, they confirm Crispo's work, as they show that there is a general tendency towards a slight increase in the Reichert-Meissl value. They also show that there is a similar tendency with the Polenske value to increase slightly. The differences observed, however, in the saponification and Valenta values are much more marked and definite. After one year's keeping the saponification values increased respectively by 9·4, 7·6, and 8·4, and after two years the value for Sample

A had increased by 16·3. The Valenta values show even greater differences—viz., −12°, −12°, and −11° C. respectively after one year, and after two years the Valenta value of Sample A shows a decrease of 28° C.

TABLE IV.

Showing the Effect of Ageing on the Composition of Samna.

Sample.	Reichert-Meissl.	Polenske.	Zeiss at 40° C.	Valenta (Acetic Acid: sp. gr. 1·060).	Saponification Value.
A.—Fresh	31·6	1·9	42·0	58	230·9
A.—After one year	32·7	2·5	42·0	45	240·3
A.—After two years	33·6	3·1	42·0	30	247·2
B.—Fresh	32·3	2·0	42·1	59	230·5
B.—After one year	33·4	2·3	42·5	47	238·1
C.—Fresh	31·9	1·6	42·2	62	227·7
C.—After one year	31·6	1·6	43·0	51	236·1

Although few in number, these results certainly show that a considerable normal increase in the saponification value takes place during the ageing of samna, with a corresponding decrease in the Valenta value. It is not proposed to discuss here the nature of the changes which take place during the ageing, but whatever they may be, they are most probably brought about by the constant high temperature experienced in Egypt during the summer months. In any case, changes that would be only of academic interest in Europe and other temperate climates become of considerable practical importance in Eastern countries, where the analyst may frequently have to decide on genuineness of butter-fat some years after its manufacture.

Syrian Samna.—The mean results of a few samples of Syrian samna, believed to be genuine, are recorded in Table V., arranged according to their ascending Reichert-Meissl values:

TABLE V.

Mean Results from Syrian Samna, arranged according to the Reichert-Meissl Values.

No. of Samples.	Reichert-Meissl Limits.	Polenske.	Zeiss at 40° C.	Valenta (Acetic Acid: sp. gr. 1·060).	Saponification Value.
2	24–25	4·3	42·6	65	227·3
2	25–26	4·8	42·5		
3	26–27	5·8	41·9		
1	27·2	5·3	43·0	60	232·8
2	28–29	6·7	42·2		
2	29–30	6·7	42·4		
1	30·6	7·9	41·5		
1	31·2	6·4	42·3		235·5

As remarked before, this kind of samna is generally believed to have its origin in the milk of sheep and goats, although some evidence has already been adduced to show that some samples at least consist entirely of sheep butter-fat. Attention has also been drawn to the extremely high normal Polenske values (4·3 to 6·4) and to the comparatively low Reichert-Meissl values (24·6 to 31·2). It will be noticed that the Polenske varies 2·1 for a corresponding variation in the Reichert-Meissl of 6·6. This variation is much larger than that found either in cow or buffalo butter-fat, and consequently the detection of small quantities of cocoanut oil in Syrian samna is a much more difficult matter than in their case. The difficulty is great when the samna is of known Syrian origin, but when its origin is unknown, the problem is at present practically without solution. Could one be certain that no goat butter-fat ever entered into the composition of Syrian samna, then the Hinks test could be employed with reasonable confidence; but even that method is unsatisfactory as being only qualitative and difficult, if not impossible, of application in the hot weather. It was chiefly in the hope of finding a method that would solve this problem that the methods recounted above were tried. The solution, however, is still to be found, and it is hoped that additional work which is now being carried out will at least indicate the direction in which the solution lies.

Indian Ghee.—The mean results of a few samples of Indian butter-fat arranged in a similar manner will be found in Table VI.

TABLE VI.

Mean Results from Indian Ghee, arranged according to the Reichert-Meissl Values.

No. of Samples.	Reichert-Meissl Limits.	Polenske.	Zeiss at 40° C.	Valenta (Acetic Acid: sp. gr. 1·060).	Saponification Value.
2	28–29	1·7	41·8	66·0	225·7
3	29–30	1·6	41·7	58·0	226·8
2	30–31	1·6	41·3	62·5	227·0
2	31–32	1·9	41·2	56·5	228·6
1	32·8	1·6	41·2	60·5	230·1

The figures, which are very similar to those published by Revis and Bolton (ANALYST, 1910, **35**, 343, and 1911, **36**, 392), indicate that the samples examined consist essentially of buffalo butter-fat; otherwise they do not call for further comment.

In conclusion, the author wishes to lay stress on the fact that the paper is only of a preliminary character. He desires to express his thanks to Mr. A. Lucas for suggesting the investigation, and for his help and advice.

THE SURVEY DEPARTMENT LABORATORIES,
CAIRO.

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NOTE ON SAMNA.

By H. DROOP RICHMOND, F.I.C.

(*Read at the Meeting, April* 2, 1913.)

The word "samna" is applied not only to the fat produced by clarifying butter, but also to the fat obtained from the tails of the long-tailed Barbary sheep or Argali (*Ovis tragelaphus*). This fat has a consistency very similar to that of butter-fat, does not easily go rancid, and makes a good edible fat suitable for cooking purposes.

Twenty years ago this fat was used in Egypt, but apparently from Mr. Trimen's paper it is no longer employed.

I cite a few analyses of commercial samna, the first two probably being the tail fat of the Barbary sheep, and the following are the analytical figures:

	Reichert-Meissl Value.	Saponification Value.
1	0·4	—
2	0·2	—
3	2·65	196·6
4	12·6	—
5	30·6	233·3
6	36·0	—
7	33·6	
8	36·2	
9	33·05	
10	34·1	
11	32·8	
12	35·2	

The first five samples do not consist of genuine butter fat, though it is probable that the others do.

* * * * *

THE COMPOSITION OF MILK.

By H. DROOP RICHMOND, F.I.C.

(*Read at the Meeting, May* 7, 1913.)

In the present communication the results obtained during 1912 in the laboratory of the Aylesbury Dairy Company are summarised.

The total number of analyses of milk was 39,179, and these are divided into several series. The largest and most important of these is that which refers to the milk as received from the farms, and the mean composition is given in Table I. The morning and evening milks are kept separate, and the numerical mean of the two milks is also given. This table, containing the results of 19,646 samples, is the most nearly representative of the true composition during 1912.

It may be considered an objection that, as the morning and evening milkings differ somewhat in quantity, it is not strictly correct to take the numerical average, but I have found that the differences between the true average and that given are so small that it is only the third place of decimals per cent. that is affected. A confirmation of this is shown by the fact that another series of samples, representing the milk sent out from the chief and branch depots, differs only by very small amounts in the monthly averages, and gives identically the same results for the yearly average; and in this series, representing 11,677 samples, the differences, which may be due to varying proportions of the morning and evening meals, are completely eliminated. Three smaller series of samples, containing 1,420, 2,341, and 1,903 samples respectively, also confirm the general average.

TABLE I.

Average Composition of Milk during 1912.

Month.	Morning Milk.				Evening Milk.				Mean.			
	Sp. Gr.	Total Solids.	Fat.	Solids-not-Fat.	Sp. Gr.	Total Solids.	Fat.	Solids-not-Fat.	Sp. Gr.	Total Solids.	Fat.	Solids-not-Fat.
		%	%	%		%	%	%		%	%	%
January ...	1·0320	12·37	3·54	8·83	1·0318	12·59	3·76	8·83	1·0319	12·48	3·65	8·83
February ...	1·0322	12·30	3·44	8·86	1·0318	12·55	3·71	8·84	1·0320	12·42	3·57	8·85
March ...	1·0322	12·19	3·34	8·85	1·0317	12·45	3·62	8·83	1·0320	12·32	3·48	8·84
April	1·0323	12·20	3·33	8·87	1·0320	12·48	3·62	8·86	1·0321	12·34	3·48	8·86
May	1·0325	12·10	3·21	8·89	1·0320	12·52	3·66	8·86	1·0322	12·31	3·43	8·88
June	1·0322	12·15	3·30	8·85	1·0318	12·60	3·76	8·84	1·0320	12·37	3·53	8·84
July	1·0317	12·20	3·45	8·75	1·0312	12·55	3·84	8·71	1·0315	12·37	3·64	8·73
August ...	1·0319	12·22	3·43	8·79	1·0315	12·75	3·95	8·80	1·0317	12·48	3·69	8·79
September ...	1·0321	12·45	3·57	8·88	1·0318	12·88	3·99	8·89	1·0320	12·66	3·78	8·88
October ...	1·0323	12·67	3·71	8·96	1·0321	13·03	4·06	8·97	1·0322	12·85	3·89	8·96
November ...	1·0321	12·93	3·97	8·96	1·0319	13·18	4·23	8·95	1·0320	13·05	4·10	8·95
December .	1·0318	12·70	3·84	8·86	1·0316	12·86	4·02	8·84	1·0317	12·78	3·93	8·85
Average ...	1·0321	12·37	3·51	8·86	1·0318	12·70	3·85	8·85	1·0319	12·54	3·68	8·86

The average percentage of fat (3·68) is lower than that found last year, and, indeed, is the lowest noted since 1900. This was due to the percentage of fat being lower than usual during the first five months of the year. As a rule it has been noticed that May and June are the two months in which the lowest percentages of fat are found, but during 1912 the months of March, April, and May were those in which the milk was poorest, and there was a distinct improvement during June. I attribute the low percentage of fat during the early months of the year to the effect of the prolonged drought of 1911 on the quantity and quality of the feeding-stuffs. It is probable that the rise in the amount of fat noticed in June is to be attributed to the unusually cold summer. As is usual, the highest percentage of fat is noticed in November, but the figure found (4·10) is higher than has been found for many years. The average difference between the morning and evening milks (0·34 per cent.) is a normal one.

During 1912 there was the usual fall in solids-not-fat during July and August, but very few cases of figures falling below 8·5 per cent. were found, and in September the solids-not-fat were normal. There was no repetition of the experience of 1911, when low solids-not-fat were frequently found from July to October.

In continuation of the tables given in former years, I give the percentages of the morning milks falling below 3·0 per cent. of fat. As a rule it is only from April to June that any appreciable number of samples containing less than 3·0 per cent. of fat is noticed; but in 1912 there were samples low in fat during the months of January to August inclusive, and consequently the table is much extended. In the evening milk and in the morning milk during the other months of the year samples yielding below 3·0 per cent. of fat were very few in number.

TABLE II.

Month.	2·9 to 3·0.	2·8 to 2·9.	2·7 to 2·8.	Below 2·7.
January	0·4	0·4	0·2	0·5
February	1·2	0·5	0·5	0·3
March	1·8	0·6	0·5	0·3
April	1·1	0·9	0·6	0·3
May	4·6	4·2	1·6	0·4
June	3·9	0·9	0·7	0·5
July	0·7	0·2	—	
August	0·8	0·2	—	0·2

During 1912 a systematic determination of the aldehyde figure, from which the percentage of proteins was calculated, and of the ash, was made, and the results of these, as well as the percentages of milk-sugar (by difference) appear below.

TABLE III.

Month.	Solids-not-Fat.	Milk Sugar.	Proteins.	Ash.
January	8·83	4·58	3·50	0·75
February	8·85	4·67	3·42	0·76
March	8·84	4·70	3·40	0·74
April	8·86	4·69	3·43	0·74
May	8·88	4·61	3·54	0·74
June	8·84	4·63	3·48	0·73
July	8·73	4·57	3·42	0·74
August	8·79	4·56	3·48	0·75
September...	8·[illegible]	4·52	3·60	0·76
October	8·96	4·60	3·60	0·76
November...	8·95	4·64	3·55	0·76
December	8·85	4·59	3·50	0·76

It is seen that, as was noticed in 1911, the percentage of proteins tends to follow that of the solids-not-fat, though the variations during 1912 are very much less.

I wish to acknowledge the great assistance given me by Mr. H. C. Huish, to whom the bulk of the above figures are due.

DISCUSSION.

Mr. J. GOLDING said that he had been asked whether any information was available as to what was the breed of the cows producing these samples. He thought that the value of the tables would be greatly increased if such information could be given, and also if, in the event of there being any herds which included, for example, Jersey cows, the figures apart from these could be given.

Mr. C. Revis said that he was afraid it would be rather difficult to obtain exact information as to the breed of the cows. His own average figure for fat in 1912 was 3·65, Mr. Richmond's average being 3·68; and his experience was the same as that of Mr. Richmond as to the months in which the fat was lowest and highest, his highest figure, as in Mr. Richmond's case, being obtained in November, for which month his average was 4·05. Such an agreement was, indeed, only to be expected when the bulks of milk dealt with were so large; but it was only fair to point out that such averages ought not to be used in judging the milk from individual farms, where, quite apart from any question of adultèration, the composition of the milk might vary considerably, according to the breed of the cows. His averages were, as he had explained last year, obtained in a somewhat different way from Mr. Richmond's, being based on analyses of samples representing large bulks of mixed milk, instead of on samples representing each consignment individually.

Mr. W. Partridge asked whether the samples examined by Mr. Richmond and those examined by Mr. Revis came from the same counties.

Mr. Revis said that the two sets of samples came largely from the same areas, which, however, were in both cases very large, extending over the whole of the Southern Counties and as far north as Oxfordshire—perhaps even to Cheshire. The figures, however, would not apply to the Northern or to the Eastern Counties.

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THE PROPORTIONATE DETERMINATION OF COCOANUT AND PALM-KERNEL OIL IN MIXTURES.

By H. R. BURNETT and CECIL REVIS.

(*Read at the Meeting, May* 7, 1913.)

At the present moment there is no doubt that cocoanut and palm-kernel oils form the main constituents of many margarine mixtures, and for this reason some means of arriving at the relative proportions of the two fats is very necessary to the technical chemist. There is no great difficulty so long as a "straight" mixture of the two is being dealt with, but as in practically all cases some other fat, usually a liquid oil, is present with them, the problem is a little more complex. There is probably no exact chemical means by which a solution may be arrived at, on account of the great similarity between these two fats; and, indeed, in all cases the result must be really arrived at by mathematical juggling with the ascertained figures and the average values which the oils give. Since these values vary between somewhat wide limits, it follows that the results are approximate, whilst, if any error be made in the determinations, the final calculation may be very far from the truth. In a paper published last year (Bolton, Richmond, and Revis, ANALYST, 1912, **37**, 183), it was shown that either fat alone could be estimated with a high degree of accuracy by means of the Reichert-Meissl-Polenske method, if carried out in a standard way, the Polenske figure being the real determining factor; but the similarity in character of the curves for cocoanut and palm-kernel oils preclude any exact knowledge of the quantities present when both are exhibited together, unless some exact idea of the quantity of the other constituents present be known. If this can be estimated, the Polenske figure determines the mixture by simple interpolation; but as usually some liquid oil, such as cottonseed, etc., has been used, for which no exact method of estimation is available, the Polenske figure does not give the necessary information.

In these cases it is only possible to fall back on the saponification value, and to arrive at the result by interpolation, assuming a certain value (which will usually be correct) for the third constituent of the mixture, when the Polenske figure becomes available as confirmatory evidence. As this process depends on the assumption that the saponification value taken for the third constituent is correct, we have devised the following simple addition to the Reichert-Meissl-Polenske process, not as an exact method of analysis, but as providing a useful check on the mathematically deduced result. We believe, however, that the figures obtained by this method are within the limits of error of such results.

In an ordinary "straight" mixture of cocoanut and palm-kernel oils, the Polenske figure will determine the proportion with at least as great exactitude as any other method. For instance, if Polenske values are plotted as abscissæ, with percentage composition as ordinates, then a straight line joining the points which represent 100 per cent. cocoanut oil and 100 per cent. palm-kernel oil respectively will include the Polenske values for all mixtures of these two. The following process is for mixtures containing other constituents:

The ordinary Reichert-Meissl-Polenske determination is made in the *standard* apparatus and by the *standard* method. The Polenske figure is obtained using $\frac{N}{10}$ *baryta.* The insoluble barium salts are then filtered off on a hardened filter-paper under pressure, and the salts washed three times with 3 c.c. of 93 per cent. alcohol (by vol.), the funnel being kept covered during filtration and washing. The paper, after all possible alcohol has been sucked out, is dropped into a wide-mouthed CO_2 flask, ten times the Polenske value in c.c.'s of 93 per cent. alcohol* (by vol.) added, and the flask boiled under a reflux condenser till the barium salts are in solution. About 5 c.c. of the hot solution are then poured rapidly into a strong test-tube (6 inches × ½ inch), which is at once closed with a stopper carrying a small bulb thermometer and an aluminium wire stirrer. The liquid is rapidly stirred, holding the tube in a good light, and the turbidity-point noticed. The liquid is then warmed till again clear, and the turbidity-point again noted. This second temperature is taken as the turbidity temperature. If desired, the tube can be fixed in a wider tube, so as to obtain slower cooling.

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* The alcohol used in these experiments had a sp. gr. of 0·8235 at $\frac{15·5°C}{15·5°C}$. Alcohol of the right strength may be obtained by placing 7 c.c. of water in a 100 c.c. flask and making up to the mark at 15·5° C. with Kahlbaum's absolute alcohol.

Working in this manner, cocoanut oil gives a turbidity temperature of 52·5° C. and palm-kernel oil 68·5° C., and mixtures of these fats give temperatures between these limits proportionate to the percentage composition. The turbidity-point is very sharp, is independent of the outside temperature, and the barium salts, on which the test depends, are quite insoluble in the cold alcohol used for the Polenske determination. The turbidity-points are also quite independent of the amounts of the two fats present in the original sample, but determine their relative percentages, and so supply the necessary information. The strength of alcohol (93 per cent. by volume) must be strictly adhered to if the values here given are to be employed. It is the most satisfactory concentration. Other oils and fats (such as are likely to be present) do not interfere. In certain cases small quantities of insoluble volatile acids distil in the Polenske method, which give barium salts insoluble in boiling 93 per cent. alcohol. In such cases a clear solution cannot be obtained. The turbid liquid is therefore poured into a long test-tube, corked, and kept upright in a water-bath at 70° to 71° C. until the solid matter has settled. The clear supernatant liquid is then poured off into the turbidity-tube and the temperature of turbidity determined. This process does not affect the results. This *permanent* turbidity, due to barium salts of acids other than those derived from cocoanut and palm-kernel oils, must be distinguished carefully from that due to palm-kernel "stearine." The barium salts of the insoluble volatile acids of this "stearine" do not dissolve in ten times the Polenske value in cubic centimetres of 93 per cent. alcohol, but the liquid becomes more turbid *immediately* the flask is removed from the water-bath.

As long as mixtures of cocoanut oil and palm-kernel oil are being dealt with, the above method gives good confirmatory evidence of the relative percentages. We have found that cocoanut oils of different Polenske values give practically identical turbidity temperatures. Samples of palm-kernel oil are not easy to obtain, and we cannot say from actual experience whether they will always give the same turbidity temperature as we have found, but probably the divergence will be small. It is to be noted that the filtration of the barium salts and their subsequent solution must be carried out *within a few hours* of the Polenske titration, as otherwise the salts become partially insoluble and the results are inaccurate.

Mixtures of the "oleines" and of the "stearines" are sometimes employed in place of the whole oils, but probably rarely. In these cases, though the turbidity temperatures do not give such accurate information, on account of the very variable composition of these products, at the same time they give most useful information as to their presence.

From samples which we have obtained, we have found that palm-kernel "oleine" (sap. value 244·8) has a turbidity temperature of 59·5° C. Palm-kernel "stearine" gives barium salts which only partially go into solution in boiling 93 per cent. alcohol, and it is necessary to make dilutions to obtain the true turbidity temperature. As dilutions with one-third and one-half the volume respectively, gave temperatures of 63·5° and 54·5° C., it follows that the true temperature for this sample of "stearine" (saponification value 247·6) was 72·5° C.

Samples of cocoanut "oleine" and "stearine" could not be obtained, but from mixtures with palm-kernel "oleine" and "stearine" it was calculated that, in the samples examined, cocoanut "oleine" gives a figure of 53° C. and the "stearine" 63° C. These figures, taken in conjunction with the Reichert-Meissl and Polenske figures, and the iodine value of the sample, will make it quite clear whether the whole oils or their products are present.

The following table gives the results obtained:

Fat.	Reichert-Meissl.	Polenske.	Turbidity Temperature. ° C.
Cocoanut oil	7·5	16·5	52·5
Palm-kernel oil	5·2	9·6	68·5
Palm-kernel "oleine" ..	7·2	12·1	59·5
Palm-kernel "stearine"	—	8·2	72·5
Cocoanut "oleine" ..			53·0 (calc.)
Cocoanut "stearine" ..			63·0 ,,
Cocoanut "oleine," 80 per cent. Palm-kernel "oleine," 20 per cent. ..	8·24	17·05	54·5
Cocoanut "stearine," 60 per cent. Palm-kernel "stearine," 40 per cent. ...	4·43	9·93	67·0

It is interesting to note that cohune oil, which is identical analytically with cocoanut oil, gives exactly the same turbidity figures.

In conclusion, we desire to thank Mr. E. R. Bolton for a valuable suggestion during the course of the work.

DISCUSSION.

Mr. E. R. BOLTON said that the authors had kindly given him opportunities of trying this method while their work was in progress, and he had found the two oils to be distinguishable by the difference indicated in the paper. According to his experience, however, it was necessary to be very careful that the alcohol was of the proper strength, and he thought it desirable to obtain the alcohol in large batches, and to make a blank determination for each. The case appeared to be like that of the Valenta test or the Maumené test, in which the results would be affected by differences in the strength of the reagents, too small to be within the limits of analysis.

The PRESIDENT suggested that it might be better to indicate the strength of the alcohol by stating its specific gravity, since confusion might possibly arise between percentage by weight and percentage by volume.

Mr. REVIS, in reply, said that the strength of the alcohol need not necessarily be 93 per cent., but it should not be greater than that, or the barium salts which palm-kernel oil yielded would not go into solution. If weaker alcohol were used, the results would still be comparable, but the widest range of temperatures was obtained when the strength was 93 per cent. by volume.

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EXAMINATION OF THE OILS FROM MANIHOT CEARA AND FUNTUMIA ELASTICA AND A COMPARISON OF THEIR PROPERTIES WITH THOSE OF LINSEED AND HEVEA OILS.

BY S. RIDEAL, D.Sc. (LOND.), F.I.C., AND L. H. D. ACLAND, B.A. (OXON.).

(*Read at the Meeting, May* 7, 1913.)

Oil from Ceara Rubber Seeds.—The seeds were about the size of a small hazel nut, had a very hard shell, and were covered with a reddish powder, which, judging from the high ash and silica content of the cake, was probably clay.

The weight of 100 seeds was 53·2 grms. The seeds are difficult to work with, not only on account of the hardness of the shells, but also because the kernels stick to the shells, making the seeds difficult to decorticate. A fairly good separation may be made by pounding the seeds in an iron mortar or passing them through a mincing-machine, and then separating them with a piece of coarse gauze through which the kernels pass. This method gave 45·3 per cent. kernel and 54·7 per cent. of shell. The separation is not complete, and the kernel contains a certain amount of sharp pieces of shell, rendering the oil-cake unfit for food unless very finely ground.

The oil is light yellow—particularly light when extracted with petroleum ether, or pressed—with a faint green fluorescence. It has a very agreeable nutty odour and taste. The content of oil was, in the kernel, 35 per cent.; in the whole seed, 15·75 per cent. The only specific test for the unrefined oil noted was the fact that when it is heated with about 1 to 2 per cent. of manganese resinate, it turns a bright pink colour. It resembles linseed oil in several respects. It throws down similar mucilaginous matter on standing; has a high iodine value, dries fairly quickly, giving a tough and distinctly elastic skin which is very white in colour; in fact, it is an excellent drying oil.

When boiled, it behaves like a sample of freshly extracted linseed oil in that it becomes very viscous. It does not get very dark under these circumstances. Treated with sulphur chloride, it gave at once a transparent gelatinous mass. On warming this, it darkens and gets rather harder, forming a fairly elastic mass. Five grms. of the oil gave 6·16 grms. of vulcanised oil.

Oil from the Seeds of Funtumia Elastica.—The seeds are small and pointed at the ends, and are roughly crescent-shaped. The husks are very thin and soft, so that decortication is unnecessary.

The weight of 100 seeds is 4·8 grms. The oil content is 31 to 33 per cent. on the whole seed. The oil is very dark in colour. It was at least dark yellow, however it was extracted; and when extracted with trichlorethylene, it was dark brown. It has a slight green fluorescence. The oil, extracted with trichlorethylene, is very difficult to decolourise. Sulphurous acid, nitric acid, and hydrogen peroxide do not affect its colour. A few drops of sulphuric acid (1 : 3) did not decolourise it appreciably. Chlorine had a slight decolourising effect. The best method is to heat the oil to about 100° to 120° C. for several hours with a mixture of fullers' earth (dried by exposure to the air, as 3 per cent. of water must be present in it for it to be effectual), mixed with recently ignited animal charcoal. The oil has a strong, bitter odour and taste, and also froths rather on "boiling." The oil deposits a little mucilaginous matter on standing; but later, after about ten days, it deposits a quantity of stearine in cold weather.

The stearine is brownish-white, and rather soft and sticky. It has the smell of the original oil, and gives a m.-p. of 47° C., melting to a clear brown liquid. Its saponification value is 184·4.

The oil is a drying oil, but not a very good one, as it gives rather a "tacky" film. When heated with 0·02 per cent. of manganese resinate, it became slightly gelatinised, but not sufficiently so to prevent it from flowing. When boiled it became rather dark in colour, and formed a very viscous liquid resembling a sample of linseed oil (freshly extracted from linseed meal) similarly treated. Heated with sulphur chloride it gave at once a brown gelatinous mass. On warming this to drive off the carbon bisulphide, it darkened and formed a very elastic but sticky substance. Five grms. of the oil yielded 6·6 grms. of substitute. The oil is very suitable for making rubber substitute.

Its bitter taste and rather high dextrorotation— + 0·7, whereas the other oils of the linseed group are all lævorotatory—suggested the presence of an alkaloid. Dragendorff's method was used in testing for the alkaloid. The extract was 0·11° per cent. of the oil taken, and was extremely bitter. This gave a yellow precipitate with potassium iodide, and with picric acid a yellow precipitate insoluble in ammonia. The alkaloid was volatile, as the water in the distillate when the trichlorethylene was being blown out of the marc with steam was distinctly bitter. This alkaloid is evidently very unstable as, on extracting the washed seeds with slightly acidulated water, a very bitter solution is obtained. This solution, however, lost its bitterness on warming with lead acetate and basic lead acetate (this mixture does not precipitate any known alkaloid). This might have precipitated a glucoside, but the solution contained no reducing sugar, and glucosides are insoluble in oils; therefore the bitterness of the oil cannot be due to a glucoside.

The seeds contain a cyanogenetic glucoside in rather a large quantity, as on adding a little very dilute acid (not more than 1 per cent.), not only was a precipitate produced in a drop of silver nitrate, but also the smell of prussic acid was very noticeable.

Methods of Analysis.—The iodine values were all done by Wij's method, allowing the flasks to stand for not less than forty-five minutes, and leaving as much iodine over as was absorbed. In the Hehner value the acidification was performed with hydrochloric instead of sulphuric acid, as potassium chloride is more soluble than the sulphate. The relative viscosity was determined at 15° C. by running the oils out of a burette, and the times for the oils compared with the time taken by water to run from the same burette. In determining the other values standard methods as given by Bolton and Revis in their book "Fatty Foods," or in the case of methods not given by them those described by Lewkowitsch, were used. The figures in the comparative table below were taken from the same sources.

The method of S. Fachini and W. Dorta (*Chem. Rev. Fett.-Ind.*, 1912, **19**, 77) for the separation of the solid and liquid acids by the solubility of the potassium salts of the liquid acids in 90 per cent. acetone was not successful. Five grms. of each of the oils were treated as in the lead-salts-ether separation, and the potash soaps extracted at the ordinary temperature with three portions of 50 c.c. each of 90 per cent. acetone. The acetone was boiled off and the soaps taken up with water, the acids liberated, and then treated with ether until the aqueous solution was clear. This method gave—

	Acetone Method. Per Cent.	Lead-salts-Ether Method. Per Cent.
Funtumia (liquid)	28·4	79·8
Ceara (liquid)	39·1	88·9

In both the lead-salts-ether and potash-salts-acetone methods for separating the saturated and the unsaturated acids, the liquid—*i.e.*, unsaturated, acids were freed from ether by distillation in an atmosphere of carbon dioxide under reduced pressure. In this way the acids are obtained in a very pure state, being neither injured by heat nor discoloured by oxidation.

The process is very simple and very rapid provided that a sufficiently fine capillary tube is used to conduct the carbon dioxide into the distilling flask.

In finding the Reichert-Meissl value, it was noticed that Funtumia oil gave no solid volatile but insoluble acids, whereas Ceara oil gives a large quantity of them.

Both of these oils are very like Hevea oil in all their properties. Not only are all three good drying oils, but also all their values are close together, as will be seen from the comparative table given below. Ceara oil differs from Hevea oil in that the oil-cake contains no cyanogenetic glucosides, whereas Funtumia oil does contain one. Both differ from Hevea oil in that they do not contain an appreciable amount of lipolytic enzymes.

Physical and chemical constants of the oils compared with linseed and Hevea oils:

	Ceara.	Funtumia.	Hevea.	Linseed.
Weight of 100 seeds ...	53·2 grms.	4·8 grms.	360 grms.	—
Oil content of kernel ...	35·0%	—	45·48%	—
Oil content of total seed ...	15·75%	31·0–33·0%	22·25%	36·40%
Sp. gr.	0·9238	0·9320	0·9258	0·931–0·938
Refractive index (15° C.) ...	1·475	1·4788	—	1·4835
Relative viscosity	13·0	14·3	—	18·0
Iodine value	135·0–137·0	138·0	138·7	192·0–200·0
Saponification value ...	189·1	185·0	191·2	190·0–195·0
Acid value	0·625	2·65	5·26	below 5·0
Hehner value	95·1	94·0	—	94·81–95·5
Reichert-Meissl	0·44	0·66	2·7	0·00
Liquid fatty acids	88·9%	79·8%	86·0%	92·5%
Iodine value thereof ...	162·5	175·5	—	—

Fat-Free Residue.

	Per Cent.	Per Cent.
Nitrogen	2·19	4·34
Proteins	14·23	27·08
Ash	17·10	5·04
Sand	0·72	0·32
Potash	2·65	1·44
Phosphoric acid ...	1·72	2·25

Other figures are given for the value of these oils. The figures for Ceara oil given by Fendler and Kuhn (*Ber. deut. Pharm. Ges.*, 1906, **15**, 426), quoted in the *Bulletin* of the Imperial Institute, 1906, p. 364, are very close to those given by us.

The slight differences between these figures and ours would be accounted for by a difference in the source of the seeds; the seeds examined by us came from British East Africa.

A. Hebert (*Caoutch. et Guttapercha*, 1912, **9**, 6232-6234) quotes the following remarkable figures for funtumia oil:

Oil content	20·0%
Sp. gr.	0·929
Acid value	1·8
Saponification value	167·6
Hehner value	94·0
Reichert-Meissl value	12·1
Iodine value	139·0
Liquid acids	60%

None of the glycerides occurring in the natural oils, especially the drying oils, have a saponification value below 170. An oil with a Reichert value of 12·1 would naturally contain a large proportion of the glycerides of the lower fatty acids, and in this case one would expect a very high saponification value—*e.g.*, cocoanut oil with a Reichert of 7·5 has a saponification value of 257. Such a large difference, a difference in kind and not only in degree, could not be accounted for by any variation in the environment in which the trees were grown. Even if his oil were not from *Funtumia elastica*, it is extremely difficult to see how his values for the Reichert and saponification values could be reconciled.

We desire to thank Mr. E. R. Bolton for his kindness in extracting samples of the seeds for us.

DISCUSSION.

Mr. R. G. PELLY said that some years ago he had occasion to examine a sample of Ceara rubber seed, with results similar to those obtained by the authors, and had come to the conclusion that it would not be practicable to use it commercially. The oil only amounted to 15 per cent. of the whole seed, and as the shells were very hard, and could not be removed without breaking up the kernels, the only practicable plan seemed to be to crush the whole seed and extract the oil by a solvent. The residual meal from the whole seeds, which was poor in proteins, could hardly be used as a cattle-food owing to the large amount of woody matter from the shells, would not fetch more than £4 per ton as a fertiliser, and, the yield of oil being so small, it seemed hardly likely that it would pay to export the seeds, although there was a fairly large supply of them. On the other hand, the use of extraction apparatus in the tropics, worked by native labour, did not seem to afford much promise of success.

Mr. L. GOWING SCOPES said he had found it necessary first to dry the seed or other material in order to extract the oil completely with trichlorethylene, although some workers in Germany appeared to have obtained satisfactory results without previous drying. It might be mentioned that practically all the trichlorethylene now available contained a small proportion of hexachlorethane, which it was almost impossible to remove, and which always remained behind with the fat. A method, however, was at present under investigation, based on the formation of a compound between the trichlorethylene and mercuric cyanide, by which it was hoped to prepare the solvent free from hexachlorethane.

Mr. CHASTON CHAPMAN remarked that if, in the case of extracted drying oils, the oil were not heated to a sufficient extent to remove the solvent completely, such traces as remained might affect the specific gravity and refractive index of the oil; whilst, on the other hand, a degree of heating sufficient for the removal of the solvent might affect the properties of the oil in other directions. He should be interested to hear whether these two oils resembled linseed oil in yielding brominated glycerides insoluble in ether.

Mr. HEHNER said that in the case of cocoanut oil extracted with carbon bisulphide he had found it impossible to remove the solvent completely by steaming.

The PRESIDENT said that at one time he used often to find, in examining rape oil extracted by petroleum ether, that a sufficient quantity of the solvent remained behind to lower the flashing-point of the oil very considerably, such oil often having a flashing-point not greatly above 200° F., whereas the normal flashing-point of rape oil was above 400° F.; and on being heated in the water-oven the oil would lose from 0·3 to 0·5 per cent. in weight. When this was pointed out to the manufacturers, they took steps to effect the more complete removal of the solvent, with the result that the flashing-point of these extracted oils was now generally normal. With regard to the "viscosity" determinations, he would like to suggest that the results should be called what he presumed they were—viz., "efflux times," the kind of viscosimeter used and the temperature being stated. The comparative figures which the authors had given for linseed oil struck him as being rather peculiar. The range of iodine value, for instance, was given as 192 to 200; but in the case of raw linseed oil it was rarely so high as 200, while lower figures than 192 were frequent. The saponification value mentioned—namely, 195—also struck him as rather high for linseed oil. For the extraction of oils in the laboratory from paints, etc., he always preferred to use ether, because this was so readily expelled, whereas petroleum spirit, unless freshly redistilled, was liable to contain substances of a less volatile character. In extracting the oil from paint, if the last traces of ether were driven off in an atmosphere of carbon dioxide, the oil was obtained in such a condition that its specific gravity, iodine value, and yield of bromides, were approximately normal if genuine linseed oil had been used.

Dr. RIDEAL, referring to the question of commercial possibilities, said that both these plants were grown to a very large extent in German East Africa and German West Africa. The funtumia seeds which they had examined came from the Cameroons, and the ceara from British East Africa; but ceara rubber was being produced on a large number of estates in German East Africa, and the quantity of seed produced on both the East and the West Coast would be very considerable in the near future. In the Malay Peninsula and Ceylon hevea seed had been largely used for fresh planting during the last few years, so that hevea oil had not yet become a commercial article; but of course there was a limit to the amount of planting that could take place. It was not likely that much more funtumia would be planted in West Africa, as hevea was beginning to be introduced there. Ceara grew very quickly, and yielded a large quantity of seed. The seed was certainly very hard, and contained only about 15 per cent. of oil; but he thought it would be possible in East Africa, in both the English and the German colonies, to extract the oil locally and use the residue as manure.

Mr. ACLAND said that they had made a few experiments with driers, the results of which were not yet published, using lead, manganese, cobalt, uranium, and vanadium linolates and resinates, and also cobalt borate, acetate, and benzoate, these last three being insoluble. They had found the drying properties of the ceara oil to be fairly good, not far removed from those of linseed oil. Funtumia oil was not quite so good as regards drying. Soya oil came next, and finally hevea oil. They had used petroleum ether in preference to ordinary ether because the latter seemed to dissolve a good deal of colouring matter, which was not the case with petroleum ether, and provided that the petroleum ether was of suitable quality, boiling at about 40° C., they had found that it was an excellent solvent for the purpose. The heating of drying oils did not do much harm provided there was no air present. It was quite simple to drive off most of the petroleum ether in a current of carbon dioxide, and the iodine value did not appear to be altered if the filtering was done through a folded paper in the oven. It was only in the case of a thin film of oil that oxygen would be absorbed quickly enough to have an appreciable effect. He did not say that filtering in the drying oven would have absolutely no effect, but he thought that any such effect would be within the ordinary errors of analysis. The oils yielded no appreciable quantity of hexabromide.

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Adulteration of Barley Meal. Sendhoff and Weinstein. (*Chem. Zeit.*, 1913, **37**, 485-487.)—Among the adulterants used in German barley meal are milling offals, consisting of all the foreign seeds and refuse sorted out at the mills; barley bran, consisting of the hulls and points of the corns separated in the preparation of pearl barley and similar products; "English dust," consisting mainly of chalk, together with refuse from all kinds of cereals, foreign seeds, coffee berries, etc.; "F meal," consisting of about 70 per cent. of calcium sulphate and 30 per cent. of sawdust; rice bran; ground maize-ear refuse; ivory-nut meal; tapioca meal. Preliminary tests may be made by sedimentation in chloroform, in which mineral impurities fall to the bottom. Chemical tests include determinations of total ash, mineral matter insoluble in hot 10 per cent. hydrochloric acid, and crude fibre by König's glycerol-sulphuric acid method. For the microscopical examination, about 2 grms. of the meal are treated in a basin with 2 to 3 c.c. of fuming nitric acid and 100 c.c. of water, and boiled for about ten minutes. After settling, the liquid is poured off and the insoluble matter collected on a filter. When this residue is treated with chloral hydrate solution (1 : 1), clear microscopic preparations of husks and foreign particles are easily obtained. Normal barley meal shows the following composition: Mineral matters, 2·5 to 3·5 per cent.; matter insoluble in hydrochloric acid, 0·5 to 1·0 per cent. (sand, etc.); crude fibre, 4·0 to 5·0 per cent.; sulphuric acid, traces; calcium oxide, 0·5 per cent. Similar average analyses are cited for the principal adulterants enumerated above, and from these, taking crude fibre as a basis, the approximate proportions of any microscopically ascertained adulterant may be calculated. J. F. B.

Reactions of Digitalis Glucosides. Digitonin. C. Reichard. (*Pharm. Zentralbl.*, 1913, **54**, 217-221; through *Chem. Zentralbl.*, 1913, I., 1368-1369.)—Digitonin, a glucoside found in digitalis leaves, has no physiological action on the heart, and has not been used in medicine; it combines with cholesterol to form a cholesteride, the reaction being quantitative. It is insoluble in water, ether, and chloroform, slightly soluble in alcohol, and dissolves in concentrated sulphuric acid to form a colourless solution which turns brown when heated, or after long exposure to the air. With cold 25 per cent. nitric acid digitonin yields a colourless solution which becomes yellow when heated: crystals form when this solution is allowed to evaporate spontaneously. A dirty, greenish-black coloration is obtained when digitonin is heated with potassium dichromate and sulphuric acid; with ammonium molybdate and sulphuric acid a light blue coloration is produced, the colour changing after a time to blue-black. Digitonin yields a green coloration, changing to violet when heated with vanadic acid, a violet-black coloration with iodic acid, and a black-green colour with tungstic acid. A solution of digitonin in acetic acid decolourises bromine water, and reduces silver and mercury salts. A characteristic reaction of the glucoside is as follows: A drop of concentrated cobalt nitrate solution is evaporated on a microscope slide until it turns deep blue in colour, when a small quantity of solid digitonin and a drop of glacial acetic acid are added; after the lapse or from twenty-four to thirty-six hours, the mixture is converted into a mass of red-coloured hexagonal crystals. W. P. S.

Composition of Honey. J. Fiehe and P. Stegmuller. (*Arbeit. Kaiserl. Gesundheitsamte*, 1913, **44**, 78-149.)—Analyses of 112 samples of honey are recorded, the samples representing the honey imported into Germany from Austria (nine samples), Hungary (four), Russia and Poland (twenty-seven), Italy (ten), Spain (seventeen), Portugal (two), North America (six), Mexico (three), South America (twenty-three), Cuba (four), Jamaica (three), and Australia (three). The quantity of water in the samples varied from 14·94 to 24·28 per cent., the average being 18·30 per cent.; in only three cases did the quantity exceed 22 per cent. The invert sugar ranged from 61·96 to 78·84 per cent., the average amount being 73·48 per cent.; 108 of the samples were lævorotatory. Four of the samples contained upwards of 10 per cent. of sucrose, the maximum quantity found being 15·40 per cent.; the least quantity found was 0·12, and the average 2·42 per cent. The acidity varied from 0·027 to 0·204 per cent., expressed as formic acid; the ash from 0·027 to 0·673, with an average of 0·150 per cent., and the phosphoric acid (as PO_4) from 0·0075 to 0·0932, the average being 0·0198 per cent. Only one sample yielded a positive reaction with Fiehe's test for invert sugar, whilst eighteen samples yielded a positive reaction with Ley's test; the presence of starch syrup was not detected in any of the samples examined, and of ninety-five samples tested for invertase eighty-seven yielded a positive reaction. Lund's tannin precipitation method was found to be of little use in ascertaining the purity of the samples. (See also ANALYST, 1912, **38**, 449). W. P. S.

Estimation of Proteins in Milk by Aldehyde Titration. W. C. de Graaff and A. Schaap. (*Ann. Falsif.*, 1913, **6**, 149-157.)—The aldehyde method described by Steinegger (ANALYST, 1906, **31**, 45) for the estimation of proteins in milk was found by the authors to be useful and trustworthy. They employed $\frac{N}{4}$ sodium hydroxide solution for the titrations, having found that under the conditions of the method the dilution is not such as to augment the acidity of the formaldehyde (*cf.*, Richmond and Miller, ANALYST, 1906, **31**, 224; 1911, **36**, 9). They deduce the factor 0·0777 for converting the aldehyde figure into percentage of nitrogen, and 0·495 for converting it into percentage of protein. These factors agree almost exactly with those given by Steinegger, but differ slightly from the factors given by Richmond (*loc. cit.*). In the case of human milk, the nitrogen factor was found to be 0·0693, and the protein factor 0·443. Butter-milk (six samples) yields aldehyde figures varying from 10·41 to 11·29; compared with the nitrogen found by Kjeldahl's process, these figures corresponded with a nitrogen factor of 0·0498 and a protein factor of 0·318. Results of further experiments showed that it is not the formation of lactic acid which causes sour milk to have a high aldehyde figure, but that the increase is due to the action of micro-organisms on the proteins present. W. P. S.

Estimation of the Bromine Absorption of Wines. T. von Fellenberg. (*Mitt. Lebensmitteluntersuch. Hyg.*, 1912, **3**, 97-99; 1913, **4**, 14-41; through *Chem. Zentralbl.*, 1912, II., 554; 1913, I., 1367-1368.)—Details of a process are given for the estimation of the bromine absorption of wines, the values obtained being of certain use in determining the quality of a wine (see below). Ten c.c. of the wine are treated with 20 c.c. of water, 20 c.c. of $\frac{N}{50}$ bromate solution (0·57 grm. of potassium bromate and 2 grms. of potassium bromide per litre), and 7 c.c. of 20 per cent. hydrochloric acid; in the case of red wines, 40 c.c. of the bromate solution are added, and the water is omitted. After the lapse of exactly five minutes, potassium iodide solution is added, and the mixture is titrated with $\frac{N}{50}$ thiosulphate solution. A blank estimation, using 30 c.c. of water, is made at the same time. The difference between the two estimations is termed "bromine absorption *A*." Another portion of 50 c.c. of the wine is then mixed with 1 grm. of calcium carbonate, and distilled until about one-third of the liquid remains in the flask; after cooling, this residual liquid is diluted to 25 c.c., and 10 c.c. of the turbid solution are treated with 4 c.c. of a 19 per cent. lead acetate solution and 6 c.c. of water. The mixture is shaken, filtered, and the bromine absorption is estimated on 10 c.c. of the filtrate; the result gives "bromine absorption *B*." Another 10 c.c. of the turbid solution are treated with 4 c.c. of basic lead acetate solution (sp. gr. 1·24) and 6 c.c. of water, filtered, and the bromine absorption estimated on 10 c.c. of the filtrate; this gives "bromine absorption *C*." The difference between "*A*" and "*B*" corresponds with the quantity of tannin and colouring matters present in the wine; also, in certain cases, to the gallic acid and various chlorophyll derivatives. The difference between "*B*" and "*C*" is due to substances which are also probably chlorophyll derivatives or complexes, whilst the residual "bromine absorption "*C*" is caused by such wine constituents as yield a red coloration with vanillin and hydrochloric acid. The following results obtained with red and white wines are recorded:

	"*A*."	"*B*."	"*C*."
White wines (37 samples)	4·53–11·47	2·62–4·85	2·04–3·55
Red wines (10 samples)	5·52–22·75	3·26–7·22	2·00–3·33

The values "*A*" and "*B*" are decreased when the wines are clarified or "sulphured," but "*C*" is not affected; whilst all the values are diminished when the wine contains added water. W. P. S.

Note by Abstractor.—The terms in which the bromine absorptions are expressed are not defined in the paper from which this abstract was taken.—W. P. S.

Estimation of Quercitin in Wine. T. von Fellenberg. (*Mitt. Lebensmitteluntersuch. Hyg.*, 1913, **4**, 1-14; through *Chem. Zentralbl.*, 1913, I., 1366-1367.) —By heating under pressure 1 kilo. of vine-shoots with 3 litres of water for thirty minutes to a temperature of 130° C., filtering the solution, and extracting the filtrate with ether, 0·17 grm. of colouring matter was obtained in the form of crystals; the latter were needle-shaped, melted at about 250° C., and gave all the reactions of quercitin (pentahydroxyflavonol). The yellow colouring matter present in wines may be extracted by means of ether, then fixed on mordanted wool, and its quantity estimated by comparison with the colour obtained under similar conditions with known amounts of the substance. It appears that only traces of quercitin occur as such in wine, and that it is quercitrin (the methyl-pentoside of quercitin) which is present. Red wine contains a brownish-red colouring matter, which may be removed by fixing it on unmordanted wool before the quercitin is dyed on to mordanted wool. Fresh grape-juice does not usually contain the yellow colouring matter, but the latter is formed gradually during fermentation, especially in the case of the juice from grape-skins. White wines do not contain more than 4 mgrms. per litre of the substance, whilst red wines contain much more, as do also both red and white wines prepared from grape-skins, etc.; the presence of more than 8 mgrms. of yellow colouring matter in a litre of white wine indicates that the wine has been prepared from grape-skin juice. This yellow colouring matter of wines probably consists of quercitrin, quercitin, and a decomposition product of a chlorophyll complex. W. P. S.

Composition of Vermouth Wines. A. Behre and K. Frerichs. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1913, **25**, 429-444.)—Results of analyses of twelve samples of vermouth wine (a stimulating liquor usually consisting of wine, to which have been added absinthe, angelica, and other aromatic substances) are recorded. The minimum and maximum figures obtained were—Alcohol, 14·45 and 16·93 per cent. by volume; total solids, 9·88 and 16·75 per cent.; ash, 0·081 and 0·258 per cent.; total acid (as tartaric acid) 0·281 and 0·473 per cent.; tartaric acid, 0·072 and 0·191 per cent.; glycerol, 0·244 and 0·669 per cent.; invert sugar, 1·77 and 14·16 per cent.; sucrose, 0 and 11·26 per cent. It appears to be exceedingly difficult to ascertain by analysis how much wine is actually present in these beverages, especially in cases where the proportion of added liquid is not excessive.

W. P. S.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Distribution of Ante-Mortem Administered Arsenic in the Human Body. J. B. Ekeley. (*J. Amer. Chem. Soc.*, 1913, **35**, 483-485.)—The following results were obtained from an analysis of the organs of a person known to have died from the effects of criminally administered arsenic. The body when analysed was in an advanced state of moist decomposition; the stomach, liver, kidneys, and heart had been removed from the body previous to burial, and traces of arsenic were found in the soil adjacent to the thin casket containing the corpse. Johnson and Chittenden's modification of Gautier's method, in which only volatile reagents are employed, was found very satisfactory. The arsenic actually found, which was only a portion of the total amount present in the body, was as follows: 60 grms. stomach contents, 0·0177 grm. arsenic; 72 grms. stomach walls, 0·0124 grm. arsenic; 821 grms. liver, 0 0598 grm. arsenic; 763 grms. intestines, 0·218 grm. arsenic; 193 grms. kidneys, 0·0355 grm. arsenic; 127 grms. heart, 0·0012 grm. arsenic; 932 grms. brain, 0·009 grm. arsenic; 18 grms. spinal cord, trace of arsenic; 100 grms. thigh, 0·0003 grm. arsenic; 82 grms. toes, 0·0002 grm. arsenic. Total, 0·1498 grm. arsenic. If the figures are calculated so as to show arsenic per 100 grms. of tissue used, by far the greater part is found to be equally divided between the kidneys and stomach walls.

These results are at variance with those of Scolosuboff (*Bull. Soc. Chim.*, (2), **24**, 124), who found most arsenic in the spinal marrow, then in the brain, next the liver, and least in the muscles; but they agree in general with those of Ludwig (*Med. Jahrbuch*, 1880) and with Chittenden (*J. Amer. Chem. Soc.*, 1883, **5**, 8). The contention of the last author that "the finding of arsenic in the brain is an indication amounting to almost positive proof that the poison was not post-mortem" would seem to be confirmed.

H. F. E. H.

Action of "Activated" Aluminium on Alkaloid Extracts and its Use in Toxicology. E. Kohn-Abrest, Rochas, and Rivera-Maltes. (*Ann. Falsif.*, 1913, **6**, 131-138.)—Aluminium which has been treated with mercuric chloride may be employed for clarifying alcoholic extracts of alkaloids, such as are obtained in the separation of alkaloids from decomposing substances in the course of toxicological analysis. The aluminium is rendered active by immersing it for three minutes in a 1 per cent. solution of mercuric chloride, and then washing it thoroughly. The aluminium may be used in the form of turnings, but small pieces of foil will be found more convenient. Preliminary experiments made by the authors show that when alcoholic solutions of alkaloids are treated with the "activated" aluminium, the alkaloid is not precipitated except in the case of strychnine, quinine, and cocaine, which are precipitated partially, whilst nicotine is removed completely from its solution. In testing viscera, etc., for the presence of alkaloids, the usual extraction is made with alcohol in the presence of tartaric acid, the alcoholic extract is filtered and treated with "activated" aluminium for twelve hours. More acid must be added if the mixture becomes alkaline during this period. The solution is then filtered, and the alkaloids are isolated from the filtrate in the usual way, and identified.

W. P. S.

Estimation of Colloids in Effluents. P. Rohland. (*Zeitsch. Chem. Ind. Kolloide*, 1913, **12**, 45-46; through *Chem. Zentralbl.*, 1913, I., 1539-1540.)—The most injurious substances in trade effluents, especially from sulphite cellulose works, are of a colloidal nature. For their estimation 50 to 100 c.c. of the effluent, which has been freed from suspended particles, are treated with 1 c.c. of a 1 per cent. solution of aniline blue, and evaporated to a syrup on the water-bath. The residue is diluted with water and filtered through a tared filter, the insoluble matter washed with water and weighed, and the amount of absorbed dyestuff deducted from the weight. The latter value is found by a colorimetric estimation of the unabsorbed dyestuff.

C. A. M.

Attempt to Estimate the Vitamine Fraction in Milk. C. Funk. (*Biochem. J.*, 1913, **7**, 211-213.)—The whole vitamine fraction (nitrogen precipitated by phosphotungstic acid in the alcoholic extract of the dried milk) from 1 litre of milk amounts to 1 to 2·5 mgrms. nitrogen. This would correspond to 10 to 30 mgrms. vitamine ($C_{17}H_{20}O_7N_2$). After the elimination of the vitamine fraction the residual nitrogen amounts to 20 to 50 mgrms. per litre of milk. This residual nitrogen represents, in all probability, allantoin; if so, then 1 litre of milk contains 0·06 to 0·15 grm. allantoin. Milk, after removal of fat by centrifuging, loses about 50 per cent. of vitamine and allantoin. The milk used in the experiments was obtained from London dairies, and the present investigation shows the ordinary chemical methods for estimating vitamines hardly suffice, and attention must be directed to colorimetric methods. The behaviour of allantoin when boiled with triketohydrindene hydrate (Ruhemann, *J. Chem. Soc.*, 1910, **97**, 2025) is of interest. Abderhalden and Schmidt (*Zeitsch. physiol. Chem.*, 1911, **72**, 87) state that when these substances are boiled together for one minute no coloration results. The authors confirm this, but state that continued boiling (two to three minutes) causes a violet coloration, which might be used as a test for allantoin. Allantoin, like vitamine, is destroyed by boiling.

H. F. E. H.

Estimation of Urea in Urine by Sodium Hypobromite. M. Krogh. (*Zeitsch. Physiol. Chem.*, 1913, **84**, 379-408.)—The reaction between sodium hypobromite and urea does not proceed completely according to the generally accepted equation:

$$CO(NH_2)_2 + 3NaOBr + 2NaOH = Na_2CO_3 + 3NaBr + 3H_2O + N_2.$$

Part of the nitrogen is converted into oxygen compounds which were not identified, and part of the carbon is only oxidised to carbon monoxide. The greater the ratio of bromine to sodium hydroxide, the more far-reaching is the oxidation. If glucose is present the method is inaccurate, as the volume of evolved gas is increased, owing to the nitrogen being completely liberated and a greater proportion of carbon being converted into monoxide. On decomposing ammonium chloride with sodium hypobromite, only part of the nitrogen is liberated, a part being converted into oxygen compounds. The concentration of the urea or ammonia does not affect the reaction, provided an excess of sodium hypobromite is present, in which case the liquid remains yellow after the reaction. Changes of temperature up to 5° C. do not affect the reaction. Certain constituents of urine other than urea are also decomposed by sodium hypobromite, with evolution of nitrogen. This source of error can be diminished, though not entirely eliminated, by precipitating the urine with phosphotungstic acid and treating the neutralised filtrate with hypobromite. For the determination of urea in urine the following method is recommended: 5 c.c. urine are titrated with a 10 per cent. phosphotungstic acid solution in $\frac{N}{2}$ sulphuric acid to determine the amount required for complete precipitation. The amount of urine which, on addition of the requisite volume of phosphotungstic acid, will yield 10 c.c., is measured into a 10 c.c. graduated flask, phosphotungstic acid added, filtered, the filtrate neutralised with 30 per cent. sodium hydroxide and decomposed with a solution of 1 c.c. bromine in 24·5 c.c. 30 per cent. sodium hydroxide + 70 c.c. water. The quantity of gas evolved $\times \frac{100}{96 \cdot 5}$ = urea nitrogen. If the hypobromite be prepared by dissolving 1 c.c. bromine in 196 c.c. 30 per cent. sodium hydroxide, the evolved gas corresponds exactly with urea nitrogen. The results obtained by this method are lower than those obtained by decomposing the phosphotungstic filtrate under pressure according to Folin: this is probably due to the presence of such substances as oxyprotein acids, which are not removed by phosphotungstic acid, nor decomposed by hypobromite, but are decomposed at high temperatures. The results obtained by the hypobromite method are more likely to be too high than too low, since phosphotungstic acid does not remove all substances decomposable by hypobromite. E. W.

Estimation of Chlorides in Urine and Blood. K. O. Larsson. (*Biochem. Zeitsch.*, 1913, **49**, 479-486.) — Bang has found that all substances occurring in urine which react with silver nitrate other than chlorides can be removed by means of blood charcoal, and that the chlorides in the filtrate can be accurately determined by Mohr's method. The author has worked out the details of the method and also its application to blood. For urine 20 c.c. are shaken with 1 grm. blood charcoal for ten minutes, filtered, and 10 c.c. of the filtrate titrated with $\frac{N}{10}$ silver nitrate, using potassium chromate as indicator. If the sp. gr. of the urine is higher than 1·025 it must be diluted with water. It is necessary that the urine be acid. The quantity of charcoal may not exceed 2·5 grms. per 20 c.c. For blood, 5 to 10 grms. are treated with about 50 c.c. of a boiling 2 per cent. magnesium sulphate solution, a few drops of 40 per cent. acetic acid added, and the mixture heated on a water-bath until coagulation is complete. After cooling, the mixture is made up to 100 c.c., shaken, and after two hours poured on to 3 grms. blood charcoal, shaken, and after a couple of minutes filtered, and the chlorine in 50 c.c. filtrate estimated by Mohr's method. The method gives exact results if 5 to 10 grms. blood are used, but if smaller quantities, 1 to 3 grms., are used, the results are too high, owing to the solutions being too dilute. This source of error can be eliminated by concentrating the liquid before titration. The coagulation of the albumin is necessary. If it be omitted, the results are 20 to 30 per cent. too low, which possibly may indicate that the salt occurs in combination with the albumin.

E. W.

ORGANIC ANALYSIS.

Estimation of Carbon by Combustion with Moist Oxygen. S. Hilpert. (*Ber.*, 1913, **46**, 949-952.)—It is known that the presence of water-vapour materially assists the combustion of difficultly combustible substances. The author has applied this principle in the combustion analysis of certain carbon compounds. Magnesium and manganese carbides containing a high percentage of carbon can only be burnt very incompletely in dry oxygen, but are readily dealt with if the current of oxygen be first passed through a washing-bottle containing water. Naturally, an efficient calcium chloride tube must be provided in front of the potash bulbs, in order to remove the moisture after combustion. Another important application of this principle is for the determination of carbon in steels. In the ordinary way this can only be burnt at the very high temperature of 1,200° C. in the electric furnace. With the use of moist oxygen, however, a temperature of 950° C. is amply sufficient in most cases to produce a complete combustion of the carbon. Only in the case of an 80 per cent. ferromanganese with 6·16 per cent. of carbon was a temperature as high as 1,050° C., with a prolonged heating, required for the analysis. The use of moist oxygen is also advantageous in certain cases in the analysis of organic substances which form hard masses of carbon or of explosive compounds. Water-vapour is very useful in the combustion of aluminium-phenyl and of diazo compounds. Nitrogenous compounds, such as hippuric acid, glycocoll, alanine, and carbazol, may be burnt completely and with accuracy without the necessity of mixing copper oxide with the substance in the boat. If it is required to determine the hydrogen in the same portion of the substance, arrangements must be made to conduct the first part of the combustion in the ordinary way, then change the calcium chloride tube, and finish up with moist oxygen. It is desirable to insert a straight tube about 5 cm. long, filled with phosphorus pentoxide, between the calcium chloride tube and the potash-bulbs, in order to absorb the last traces of moisture. Finally, the moist oxygen gives very good results when employed for the combustion analysis of substances such as phenyl-phosphoric acid, in which the carbon becomes coated with fusible ash. In the ordinary way only a very careful mixing of the substance with much copper oxide will give accurate results, but with moist oxygen the substance is readily burnt, leaving a residue of perfectly white metaphosphoric acid.

J. F. B.

Experiments on the Direct Estimation of Caoutchouc by Titration with Bromine. F. Kirchhof. (*Gummi. Zeit.*, 1913, **27**, 9; through *Chem. Zentralbl.*, 1913, I., 1467-1468.)—Five grms. of extracted raw Para rubber are dissolved in a stoppered bottle in about 200 c.c. of carbon tetrachloride, and treated with an excess of $\frac{N}{8}$ bromine solution (in carbon tetrachloride). After a time of action varying from 4 to 12 hours, with repeated shaking, a concentrated aqueous solution of potassium iodide is added, and after complete reduction of the excess of bromine, the separated iodine is titrated with $\frac{N}{10}$ thiosulphate, using starch as an indicator. It was found that the reaction afforded no sharp end-point for the titration, since the decolourised solution always turned blue again on standing. Further, it was found that the quantity of bromine absorbed by the caoutchouc depended largely on the time allowed for its action. The observed values ranged from 94·4 to 117 per cent. calculated as $C_{10}H_{16}$. The quantity of hydrobromic acid formed varied according to the purity and dryness of the reagents, the temperature, concentration, time, and other conditions of the action. No better results were obtained with vulcanised rubber. J. F. B.

Method for the Estimation of the Nitrogenous Constituents of Raw Caoutchouc. A. Tschirch and W. Schmitz. (*Gummi. Zeit.*, 1912, **26**, 2079-2081; through *Chem. Zentralbl.*, 1913, I., 1467.)—From raw Para rubber the authors obtained 2·9 to 3·1 per cent. of a residue insoluble in pentachlorethane, which contained 11 to 12·6 per cent. of nitrogen. The raw rubber itself contained 0·3571 per cent. of nitrogen, of which 0·345 per cent. was found in the residue from the pentachlorethane extraction. After deducting the ash, the nitrogen in the residue amounted to 14 to 15·6 per cent., which is very nearly that contained in a pure protein. Nevertheless this residue does not show the well-defined reactions characteristic of proteins, so that the authors are inclined to think that the nitrogenous constituents of crude rubber may not be true proteins. J. F. B.

Estimation of the Viscosity of Rubber Solutions. J. G. Fol. (*Chem. Weekblad.*, 1913, **10**, 154-174; through *Chem. Zentralbl.*, 1913, I., 1369.)—The author reviews various methods which have been proposed, particularly that of Schidrowitz and Goldsbrough (ANALYST, 1909, **34**, 112), and gives the results of his own experience as to the estimation of the viscosity of rubber solutions. He comes to the conclusion that Ostwald's capillary viscometer is the most suitable apparatus for the purpose, but the diameter of the capillary must not be too small. The viscosity is affected if the solution is shaken violently during its preparation, and there is a considerable difference in the relative viscosities of solutions of equal concentration in which the rubber is dissolved completely and incompletely, respectively. The viscosity also varies with the age of the solution, and the absolute viscosity, but not the relative viscosity, of Hevea rubber alters with the temperature. In the case of the more concentrated solutions, the time of efflux diminishes while successive determinations are being made. The following method is recommended: The rubber is cut up into small pieces, dried *in vacuo* over sulphuric acid, and portions of 0·025, 0·5, and 1·0 grm. are weighed into brown glass bottles. After 100 c.c. of pure benzene have been added to each bottle, the contents are shaken twice a day for three days, then filtered through glass-wool, allowed to stand for another day, and the viscosity then estimated at 30° C. in an Ostwald's capillary viscometer of standard dimensions; three readings are taken for each solution, and the concentration of the same is found by evaporating portions and weighing the residues. A viscosity curve is now plotted with a standard scale (1 c.c. on the x-axis = 2 viscosity units; 1 c.c. on the y-axis = 0·1 per cent. concentration). The viscosity number, which serves as a measure of comparison between different rubbers, is represented by the area (in sq. cm.) between the co-ordinates, the viscosity curve, and the ordinate corresponding with 1 per cent. concentration. W. P. S.

Analysis of Mixtures of the Lower Fatty Acids. K. Langheld and A. Zeileis. (*Ber.*, 1913, **46**, 1171-1177.)—An accurate method for the separation of the lower fatty acids is required, particularly in the investigation of the group of monoamino-acids formed by the hydrolysis of proteins. The authors have devised a method of fractional moist combustion by means of chromic acid, which they have developed for the analysis of mixtures containing four typical acids of the series—viz., acetic, isobutyric, isovaleric, and methyl ethyl acetic acids. Using chromic and sulphuric acid mixture, it is found that the oxidation of the acids may be split up into three stages, corresponding to the temperatures of 65°, 100°, and 170° C., and that the branch-chains of the iso-acids are oxidised at the lower temperatures. The following table shows the number of molecules of carbon dioxide split off from the type acids at various temperatures:

	At 65° C.	At 100° C.	At 170° C.
Acetic acid	—	—	2
Isobutyric acid	1	1	2
Isovaleric acid	2	1	2
Methyl ethyl acetic acid	1	1	3

The determination of the carbon dioxide produced at these three stages and the total acidity of the mixture give four data, from which the percentage of each of the four acids in the mixture may be calculated. Thus, let a, b, and c, represent the weights of carbon dioxide produced at the respective temperatures above, and let t represent the number of c.c. of $\frac{N}{10}$ alkali required to neutralise the total acidity of the mixture, then:

$$\text{Acetic acid} = \left(t - \frac{10000 \cdot b}{44}\right) . 0{\cdot}006 \text{ grm.}$$

$$\text{Isobutyric acid} = \frac{88}{44} . \left(2b - a - c + 2 . \frac{44.t}{10000}\right) \text{ grm.}$$

$$\text{Isovaleric acid} = \frac{120}{44} . (a - b) \text{ grm.}$$

$$\text{Methyl ethyl acetic acid} = \frac{102}{44} \left(c - 2 \cdot \frac{44.t}{10000}\right) \text{grm.}$$

The method is carried out in the following manner: The oxidation is performed in the apparatus devised by Messinger for moist combustions. The acid mixture is first accurately titrated with $\frac{N}{10}$ alkali, and a quantity of the neutralised solution, equivalent to 60 to 70 c.c. of $\frac{N}{10}$ alkali, is taken for the combustion. The liquid is concentrated to about 25 c.c. and transferred to the combustion flask. To this are added, whilst cooling in a freezing mixture, 100 c.c. of concentrated sulphuric acid, and the liquid is allowed gradually to assume the temperature of the atmosphere. Meanwhile, air freed from carbon dioxide is passed through the apparatus, and when the latter is free from carbon dioxide the absorption flask is connected up. A solution of 7 grms. of chromic acid in 25 c.c. of water, mixed with 100 c.c. of concentrated sulphuric acid, is then admitted through a dropping funnel, and the reaction flask is placed in a water-bath at 65° C., which is kept at this temperature until the evolution of carbon dioxide at this stage is finished; this requires about thirty minutes. The apparatus is then cleared out by a current of air free from carbon dioxide, and the absorption vessel is weighed. The same operations are repeated at the other two stages of the combustion at temperatures of 100° and 170° C. respectively. It is to be noted that, for the complete oxidation of acetic acid and propionic acid, about three hours are generally required. The results are sufficiently good to afford very useful indications not readily obtainable by older methods, and the process possesses the advantage that only small quantities of the material are required for the analysis. J. F. B.

Detection of Volatile Fatty Acids. H. Agulhon. (*Bull. Soc. Chim.*, 1913, **13**, 404-408.)—When the aqueous solution of the sodium salt of a fatty acid is treated with copper sulphate and then shaken with certain organic solvents, a whole or part of the copper salt of the fatty acid may be extracted, the proportion removed being a function of the molecular weight of the fatty acid. For example, none of the usual solvents extract the copper compound in the case of sodium formate or acetate. With the propionate there is a slight blue coloration on extracting the solution with ethyl acetate, but no coloration with ether, chloroform, or benzene. Sodium butyrate, however, gives an intense blue coloration with ethyl acetate, ether, chloroform, and amyl alcohol, but not with benzene or toluene if the proportion of the sodium salt did not exceed 1 or 2 per cent.; whilst in the case of sodium valerate or caproate all the solvents remove the whole of the copper salt. Similar differences are to be observed in the behaviour of the iron salts of the different fatty acids. In applying the method, the liquid, which must not contain more than 2 per cent. of fatty acid, is exactly neutralised with a standard solution of sodium hydroxide, with phenolphthaleïn as indicator, and 2 c.c. of the solution are taken for each test. About 1 c.c. of ether is added, and then, drop by drop, with continual shaking, a 2 per cent. solution of copper sulphate. A blue coloration of the ether indicates a butyrate, valerate, or the salt of a higher fatty acid. The test is now repeated with 1 c.c. of benzene, and if the latter remains colourless, butyric acid is probably present, whilst a blue coloration denotes the presence of valeric acid or a higher fatty acid. If in the first test the ether remained colourless, the test is repeated with ethyl acetate and a dilute (less than 5 per cent.) solution of ferric chloride, when a yellow coloration of the solvent indicates propionic acid, whilst with acetic acid it will still be colourless.

C. A. M.

Detection of Formic and Acetic Acids. L. Bonnes. (*Bull. Sci. Pharmacol.*, 1913, **20**, 99-101; through *Chem. Zentralbl.*, 1913, I., 1364.)—Formic and acetic acids may be detected in such substances as glycerol by distilling about 4 c.c. of the sample with an excess of sulphuric acid, neutralising the distillate by the addition of an excess of calcium carbonate, and evaporating the mixture to dryness. The residue is then subjected to dry distillation, and the distillate is collected in a small quantity of water. A portion of this solution is tested for the presence of aldehyde with Ley's rosaniline bisulphite solution, and another portion for acetone by the sodium nitroprusside test. If Ley's solution gives a positive reaction, formic acid is present in the sample, although a negative reaction may result with the nitroprusside test; the presence of acetic acid is denoted by a positive reaction with nitroprusside and a negative reaction with Ley's solution. Positive reactions with both tests indicate the presence of the two acids together. W. P. S.

Pine-nut Oil. M. Adams and A. Holmes. (*J. Ind. and Eng. Chem.* 1913, **5**, 285-287.)—The kernels of the nuts of the pine-nut tree, *Pinus monophylla*, which grows on the eastern side of the Sierra Nevada Mountains, yield an oil resembling olive oil, with a bland aromatic taste. The freshly extracted oil is light yellow, but the colour gradually fades. A sample of the nuts yielded 12·4 per cent. of oil (16·2 per cent. on the kernels). This oil melted at −15° C. and distilled without decomposition at 305° C. under a reduced pressure of 60 mm. It gave the following values: Refractive index at 15° C., 1·4733; at 40° C., 1·4543; saponification value, 189·31; iodine value (Hübl), 107·9; iodine value of "liquid" fatty acids, 117·1; and glycerol, 9·2 per cent. No phytosterol could be detected. The fatty acids consisted of oleic acid, with small amounts of stearic (1 per cent.), palmitic, lauric, and linolic acid. No linolenic acid was found, but there was apparently some other unsaturated fatty acid, which was not identified. C. A. M.

New Method of Estimating Glycerol. S. H. Bertram. (*Chem. Weekblad.*, 1913, **10**, 237-239; through *Chem. Zentralbl.*, 1913, I., 1543.)—The solution containing the glycerol is treated with excess of $\frac{N}{2}$ potassium hydroxide solution and 10 per cent. copper sulphate solution until a permanent precipitate is obtained. The liquid is then acidified with acetic acid, an excess of potassium iodide solution added, and the separated iodine titrated with $\frac{N}{10}$ thiosulphate solution. Each molecule of sodium thiosulphate corresponds to two molecules of glycerol. (*Cf.* Muter, ANALYST, 1881, **6**, 41.) C. A. M.

Heat Test. I.—Guncotton. II.—Nitroglycerine and Cordite. III.—Theoretical. A. C. Egerton. (*J. Soc. Chem. Ind.*, 1913, **32**, 331.)—Several weak points in the Abel heat test are pointed out, and a new test is described, in which the paper is replaced by a drop of α-naphthylamine and sulphanilic acid in acetic acid, or of dimethylaniline in acetic acid. The drop, of definite surface, is contained in a small cell placed in a compartment attached to the ordinary heat-test tube. With the paper indicator, a guncotton continuously tested gives first (1) a fall and then (2) a rise of heat test. Differing from Smart and Robertson (ANALYST, 1910, **35**,

171) who regard this as due to (1) nitric peroxide retained by the heated guncotton, and (2) to a drying of the test paper, the author attributes it to the formation of nitric acid from the peroxide and the moisture in the guncotton; at first (1) there is sufficient moisture, and the test falls; but (2) on continued heating of the guncotton it loses moisture, less nitric acid is formed, and the test rises. The fall is less marked with the drop-test. Experiments on the variation of moisture during the test and on the effect of evacuation are given in support of this theory. A continuous test is stated to give much more valuable results than a single one. With nitroglycerine and cordite the presence of several test-papers or drops produces little alteration in the test, although the amount of nitric peroxide absorbed by them is not negligible; the setting up of an equilibrium between the partial pressure of the nitric peroxide and the dissociation pressure of the explosive at the test temperature is assumed to account for this. The equilibrium pressure is stated to be a more certain measure of stability than the Abel test, and it is maintained that the curve given by a continuous test affords a satisfactory indication of the tendency of an explosive to decompose. O. E. M.

Analysis of Mixtures of Hydrogen with Methane, Ethane, Propane, and Isobutane. P. Lebeau and A. Damiens. (*Bull. Soc. Chim.*, 1913, **13**, 366-372.)—The paper describes the authors' work with mixtures of hydrogen, methane, ethane, and propane (Analyst, 1913, 121), and, in addition, shows how, by a modification of their method, mixtures containing these gases together with isopropane may be analysed. The hydrogen and methane are first separated in the manner already described (*loc. cit.*), and separately estimated in any convenient manner. The residual liquid—consisting of ethane, propane, and isobutane—is then allowed to attain a temperature of −127° C. by substituting for the bath of liquid air a bath of light petroleum cooled to −127° C., and kept at, or about, that temperature by being enclosed in a Dewar vessel, into which liquid air is introduced drop by drop, so that it always stands at a marked level. The temperature of the petroleum bath is determined by the vertical height between its base and the surface of the liquid air, and is measured by means of a calibrated thermo-couple. The temperature may rise to −123·5° C. without detectable error. With the temperature between −127° and −123·5° C., the pump is set in operation and the gas, which contains all the ethane and some propane, is removed and analysed eudiometrically. The residual liquid, which contains all the isobutane and the rest of the propane, is then allowed to assume the gaseous state and analysed similarly to the other binary mixtures. G. C. J.

Colour Reactions of Certain Indole Derivatives and their Significance with regard to the Glyoxylic Reaction. A. Homer. (*Biochem. J.*, 1913, **7**, 116-126.)—The formation of coloured condensation products from indole derivatives and certain aldehydes necessitates the use of condensing agents. The reaction takes place between the −NH group of the indole nucleus and the −CHO group of the aldehyde. The evidence adduced in this paper is favourable to the Adamkiewicz reaction being primarily a formaldehyde reaction. The formaldehyde condensation product of tryptophane gives the colour test directly with concentrated sulphuric acid without the addition of an oxidising agent or of glyoxylic acid or of formaldehyde. A trace of formaldehyde without the addition of an oxidising agent gives the colour reaction with indole derivatives. When formaldehyde is used in testing for indole derivatives, the purple colour is often masked on account of further reactions taking place between the indole substance and the trioxymethylene formed from formaldehyde by the condensing power of the concentrated acid. In Hopkins and Cole's modification of the test, glyoxylic acid reacts by virtue of its decomposition into formaldehyde, and for one of the two following reasons is the best reagent to use for the reaction: (*a*) The formaldehyde is liberated in such small quantity that the formation of trioxymethylene does not take place to any appreciable extent; (*b*) the formaldehyde so liberated is in the nascent state, and therefore more reactive. In the case of tryptophane the colour test is often performed with formaldehyde in the presence of an oxidising agent. The part played by the latter is primarily to produce some oxidation product of tryptophane—*e.g.*, indolealdehyde. The reaction under these conditions is of a complex nature, as besides the effects described above, it involves the formation of coloured substances by the action of the concentrated acid on (*a*) indolealdehyde and tryptophane; (*b*) indolealdehyde in the presence of an oxidising agent; (*c*) indolealdehyde and tryptophane in the presence of an oxidising agent; (*d*) indolealdehyde and tryptophane in the presence of formaldehyde.

H. F. E. H.

Application of the Dimethyl Sulphate Test for Determining Small Amounts of Petroleum or Asphalt Products in Tars. C. S. Reeve and R. H. Lewis. (*J. Ind. and Eng. Chem.*, 1913, **5**, 293-295.)—Valenta stated (ANALYST, 1906, **31**, 202) that aromatic hydrocarbons were miscible in all proportions with dimethyl sulphate, whereas open-chain hydrocarbons were entirely insoluble in that reagent. Chapin has shown (*U.S. Dept. Agric., Circular* 167) that open-chain hydrocarbons show varying degrees of solubility in dimethyl sulphate, and that when a mixture of aromatic and open-chain hydrocarbons is shaken with the reagent, a varying, though usually small, amount of the aromatic hydrocarbons remains dissolved in the upper (paraffin) layer. Nevertheless, he pronounced the test of value, and Sommer has attempted to apply it to the determination of asphalt in tar-asphalt paving mixture. Sommer (*J. Ind. and Eng. Chem.*, 1910, **2**, 181) showed that the distillates from native bitumens (largely polymethylenes) were insoluble in dimethyl sulphate, and he proposed to distil the tar-paving mixture to coke, and to test 4 c.c. of the distillate by shaking with 6 c.c. of the reagent in a graduated cylinder. In the distillate from a mixture containing 80 per cent. asphalt, he found 60 per cent. insoluble, whilst a 20 per cent. mixture (the poorest he tried) gave 15 per cent insoluble. The present authors failed to get any evidence of the presence of asphalt when applying Sommer's method to 10 per cent. paving mixtures, and consider that this is not surprising, as a distillate obtained in Sommer's manner might be expected to contain a much higher proportion of aromatic hydrocarbons than the original mixture. As some current specifications provide for smaller proportions than 10 per cent., and since the control of deliveries to such specifications is extremely difficult in the present state of our knowledge, the test was further investigated and modified as follows: The sample (100 c.c.) is distilled, and the fractions distilling below 315°, between 315° to 350°, and between 350° to 375° C., are collected separately. The two higher fractions are then measured and submitted to Sommer's test of shaking 4 c.c. with 6 c.c. of dimethyl sulphate. Experiments with asphalts of various origin show that the fraction distilling below 315° C. is unlikely to contain a detectable trace of asphalt, even when the product is a high-grade one. The modified method serves to detect asphalt in mixtures containing as little as 3 per cent. It can only be used for the approximate estimation of the amount of asphalt if the grade and character of the asphalt is known, for different asphalts may yield anything from 30 to 50 per cent. of distillate below 375° C. The proportion of the distillate insoluble in dimethyl sulphate is seldom less than 80 per cent., and never more than 90 per cent. Provided the character of the asphalt is known, the figures in the paper show that its amount might be estimated with an error not greater than ± 20 per cent., or that failure to find as much as 8 per cent. where 10 per cent. was specified would be tolerably strong evidence that the sample was not up to specification. Until a more exact method is forthcoming, the authors think that their test, in conjunction with a volatilisation test to determine the character of the material which has been fluxed with a tar, will prove useful. Failing more exact methods, they suggest that the chemist's duty in seeing that specifications are complied with would be more easily fulfilled if the grade and character of the asphalt product and tar to be used were specified as well as their relative proportions. G. C. J.

Hydrolysis of Organic Phosphorus Compounds by Dilute Acid and Dilute Alkali. R. H. A. Plimmer. (*Biochem. J.*, 1913, 7, 72-80.)—Plimmer and Scott (*J. Chem. Soc.*, 1908, **93**, 1699) have shown that the phosphoproteins may be distinguished from other organic phosphorus compounds owing to the fact that the phosphorus of the former may be completely separated as inorganic phosphate in twenty-four hours by treatment with 1 per cent. caustic soda at 37° C. Under these conditions soda does not decompose the other natural organic phosphorus compounds. The action of dilute acid and alkali from normal to twice normal strength has been studied upon glycerophosphoric acid, phytic acid, ethyl phosphoric acid, hexosephosphoric acid, nucleic acid, hydroxymethyl-phosphoric acid, and phosphoprotein. The analyses of phosphoric acid were made by precipitation with ammonium magnesium citrate and conversion into magnesium pyrophosphate. Total phosphoric acid in the solution was estimated in a separate sample by Neumann's method (*J. Physiol.*, 1906, **33**, 439). Inorganic P_2O_5 not being precipitated in the presence of phytic acid, was estimated by precipitation with ammonium molybdate. Nitric acid was used for the acid hydrolyses. Glycerophosphoric acid is slowly hydrolysed by dilute acid, but is not attacked after eighty-six days by $\frac{N}{2}$ alkali. Ethyl phosphoric acid is also stable to alkali, but is hydrolysed by acid more slowly than glycerophosphoric acid. Phytic acid is the most stable of all the organic phosphorus compounds, complete hydrolysis not being effected even by $\frac{N}{2}$ nitric acid in seventeen days at 75° C. $\frac{N}{1}$ alkali has no action upon phytic acid after thirty-two days at 75° C. Hexosephosphoric acid is easily hydrolysed by both acid and alkali. Nucleic acid is completely hydrolysed by $\frac{N}{1}$ hydrochloric acid in eight days at 75° C., and about half hydrolysed by $\frac{N}{1}$ caustic soda in thirty-two days. Hydroxymethyl-phosphoric acid is not hydrolysed by dilute acid at 75° C. Stability to alkali is a property of the esters of phosphoric acid, while hexosephosphoric acid and phosphoprotein are so different in their behaviour to alkali that some difference in their constitution from that of the esters must exist. Nucleic acid, since it is hydrolysed by both acid and alkali, seems to occupy an intermediate position between the stable esters and the unstable hexosephosphoric acid. H. F. E. H.

Investigation of Phytin. R. H. A. Plimmer and H. J. Page. (*Biochem. J.*, 1913, **7**, 157-173.)—Phytin is generally regarded as the calcium magnesium salt of inositol phosphoric acid (phytic acid). Commercial phytin, prepared from oil-cake by extraction with 0·5 to 1 per cent. of hydrochloric acid, was used almost entirely, but for comparison neutral calcium phytate was prepared from bran. The large amount of experimental work described may be summarised as follows: (1) Inorganic phosphates in phytin can be readily estimated by precipitation with ammonium molybdate in seminormal nitric acid at room temperature. (2) The calcium content of phytin cannot be estimated by precipitation as oxalate, but is easily ascertained by precipitation as calcium sulphate. The magnesium content of phytin can then be estimated as magnesium pyrophosphate. (3) There is great difficulty in removing the calcium from phytin in the preparation of phytic acid. (4) The yield of inositol, obtained in the hydrolysis of phytic acid by acids, is not quantitative; it is probable that there is another organic constituent present in phytin. H. F. E. H.

Density and Solution Volume of some Proteins. H. Chick and C. J. Martin. (*Biochem. J.*, 1913, **7**, 92-96.)—The authors have previously pointed out (*Zeitsch. Chem. Ind. Kolloide*, 1912, **11**, 102) that there was shrinkage in volume and corresponding increase in density when caseinogen formed colloidal solution. Lüdekings (*Wied. Ann.*, 1888, **35**, 552) stated that gelatine in the dry state, with a density of 1·412, gave a value of 1·9 when calculated from jellies (14 to 35 per cent. gelatine). In addition to caseinogen, crystalline serum albumin, serum globulin (pseudoglobulin), and crystalline egg albumin, were investigated. The results obtained were as follows:

Protein.	In Solution.			Dry State.
	Concentration of Protein per Cent. by Weight.	Density of Solution.	Calculated Density of Protein.	Density of Protein.
Caseinogen ...	7·85	1·0241	1·390*	1·318
Egg albumin ...	14·60	1·0401	1·359	1·269
Serum albumin	22·15	1·0647	1·378	1·275
Serum globulin	15·33	1·0428	1·365	1·279

Corrected for presence of sodium.

From the first and second columns of figures may be calculated the excess degrees of gravity (water 1,000) due to 1 grm. of protein per 100 c.c., and if this be done, the values are 3·07, 2·74, 2·92, and 2·79. Figures are also given showing the variation in density of caseinogin (sodium caseinogenate) solutions of different concentrations, the value rising from 1·412 in 9·39 per cent. solution to 1·465 in 1 per cent. solution. The density in solution of these proteins is thus from 5 to 8 per cent. in excess of their densities in the dry state, showing the extent of shrinkage in volume taking place on entering colloidal solution. H. F. E. H.

Estimation of Water in Bleached Shellac. H. Wolff. (*Farbenzeit.*, 1913, **18**, 1389-1390; through *Chem. Zentralbl.*, 1913, I., 1549.)—A large proportion of water depresses the melting-point of bleached shellac to such an extent that a sample may fuse even at 60° C. Under these conditions the mass will retain the water, so that long-continued heating (with risk of loss of volatile constituents) at 100° C. is required to expel it completely. For an accurate estimation of the water, the shellac is divided into very small fragments, and 3 to 5 grms. of an average sample are rapidly pulverised and allowed to stand for several hours in a tared dish. This is next heated at about 50° to 60° C. (care being taken that the mass does not fuse) for about an hour, after which the temperature is gradually raised (about 5° C. in thirty minutes) to the melting-point, and is finally maintained somewhat below 100° C. for fifteen to thirty minutes, then cooled and weighed. C. A. M.

Estimation of Rosin in Soap. A. A. Besson. (*Chem. Zeit.*, 1913, **37**, 453-454.)—To obtain accurate results in the estimation of rosin by Twitchell's method, the esterification must be repeated. From 2 to 2·5 grms. of the fatty acids from the soap are dissolved in 20 c.c. of absolute alcohol, the solution saturated with dry hydrochloric acid gas, and the flask closed and allowed to stand for thirty minutes to an hour. A little pumice and 150 c.c. of water are then introduced, and the liquid boiled until there is a sharp separation of the esters and rosin acids, after which the contents of the flask are transferred to a separating funnel and shaken with ether in the presence of a little methyl orange, and left for two hours. The acid layer is now drawn off, and the ethereal solution shaken with 100 c.c. of alcoholic alkali (potassium hydroxide, 5 grms.; alcohol, 50 c.c.; water, 500 c.c.), with phenolphthaleïn as indicator, and the alkaline extract run off after two hours, this extraction being repeated. The united alkaline extracts are acidified (methyl orange as indicator), shaken with 100 c.c. of ether, and allowed to stand for two hours, after which the aqueous layer is run off, and the ethereal solution evaporated in a tared Erlenmeyer flask. The residue is weighed and mixed with 2 grms. of fatty acids, the behaviour of which in Twitchell's test is known, and the mixture dissolved in 20 c.c. of alcohol, esterified and extracted with ether as described. After removal of the acid layer, 100 c.c. of water are added, and the aqueous layer neutralised with alkali, with methyl orange as indicator, and left for two hours, after which it is run off. Finally, the ethereal solution is titrated with $\frac{N}{2}$ or $\frac{N}{10}$ alcoholic potassium hydroxide solution, with phenolphthaleïn as indicator. If great accuracy is required, the extraction with alkali must be repeated again, the results being then about 0·3 per cent. higher. C. A. M.

Determination of Sucrose in Cane Molasses by the Double Polarisation Method, using Invertase and Acid as Hydrolysts. J. P. Ogilvie. (*Intern. Sugar J.*, 1912, **14**, 89-93.)—In a previous paper (ANALYST, 1911, **36**, 120) on the determination of sucrose in beet products, it was shown by the author that if the selective hydrolyst invertase be used to effect inversion, the results are distinctly higher than those obtained by the ordinary procedure, in which the direct polarisation is taken in a solution that is alkaline owing to the presence of the basic lead acetate used for defecation. It was pointed out that if the direct polarisation be taken in an alkaline solution, then the difference between the direct and inversion readings is not due solely to sucrose, but to some extent to the presence of optically active non-sugars, such as asparagine, aspartic acid, glutamine, and glutamic acid, which have a lower rotation in alkaline than in acid solution, results that are too low to the extent of 0·5 to 1·5 per cent. thus being obtained. Hence, so as to insure that the difference between the direct and inversion readings shall be entirely due to the sucrose hydrolysed, the direct as well as the inversion reading must be taken in an acid solution, and it was shown that when this is done the results are in accordance with those found by invertase, which only hydrolyses the sucrose (and raffinose also, if present) without affecting the optically active amino-acids.

The investigation has now been extended to cane molasses, using the same method of procedure, and especially comparing the results found by the invertase method with those given by acid hydrolysis when the sucrose is calculated from the alkaline and acid polarisations. Operating upon Cuban, Egyptian, and Javan molasses, and upon "American syrup," results were obtained which lead the author to the following conclusions: (1) That if in the acid hydrolysis method the generally employed procedure of using the alkaline direct polarisation be adopted, the results may be too high to the extent of 0·4 to 1·3 per cent., according to the impurity of the product. (2) That if in the acid hydrolysis method the neutral direct polarisation in place of the alkaline be used, the results are nearer the truth, but may again be too high, especially when much reducing sugars are present. (3) That if in the acid hydrolysis method the hydrochloric acid direct polarisation, as recommended by Andrlik, be used, then correct results are obtained.

It is further pointed out that whereas in the case of beet molasses it is the optically active amino-acids that vitiate the polarimetric readings, with cane molasses it is the reducing sugars, which are always present to a more or less extent. In the presence of basic lead acetate the dextro-rotation of a cane molasses solution is considerably increased, owing to the effect of the reagent on the lævulose; while after inversion, the solution being acid, this action is eliminated, and the opposite effect obtains—namely, the lævo-rotation is increased. Thus the difference between the direct and the inversion readings is not solely due to hydrolysed sucrose, and an important source of error is introduced. Concluding, it is pointed out that both readings must be made under the same conditions of acidity by using the hydrochloric or sulphurous acid methods. When this is done, the agreement with the invertase process is very close, and well within the limit of experimental error.

J. P. O.

Use of Sulphurous Acid in making the Acid Reading in the Double Polarisation Method of Determining Sucrose. J. P. Ogilvie. (*Intern. Sugar J.*, 1912, **14**, 624-630.)—In reply to criticisms on the sulphurous acid process of making the acid direct reading in the double polarisation (Clerget-Herzfeld) method of determining sucrose in sugar-factory products, it is pointed out by the author that Pellet's original procedure does not give correct results, and that a modification of it must be used to obtain figures equal to those found by hydrochloric acid (Andrlik). This modification is carried out by saturating the 50 c.c. of defecated solution contained in a 100 c.c. flask with sulphur dioxide, or in making up the volume to 100 c.c. with an aqueous sulphurous acid solution containing about 7·5 grms. of sulphur dioxide in 100 c.c. Figures are given indicating that the results thus found are equal to those obtained when hydrochloric acid is used, and that when a lower concentration of sulphurous acid solution than that specified is used, the results do not agree with those obtained with hydrochloric acid. It is claimed that this method of working with sulphurous acid in excess solves the problem of making the acid direct reading in the case of beet molasses (see previous abstract), and obviates the error due to optically active substances other than sucrose. Moreover, in the case of hydrochloric acid and urea (Andrlik) the rate of inversion is comparatively rapid, with sulphurous acid it is reasonably slow, and the readings remain quite constant in the case of beet molasses for about thirty minutes.

For the determination of sucrose in beet molasses by the double polarisation method, using the modified sulphurous acid direct reading, the procedure now recommended is as follows: 200 c.c. of a normal sugar weight solution of the sample, defecated with basic lead acetate and filtered, is prepared. For the direct reading 50 c.c. of this solution are transferred to a 100 c.c. flask, saturated with sulphur dioxide, the flask being immersed in cold water, and made up to volume with water at standard temperature. Or, instead of saturating the assay liquid with sulphur dioxide, the 50 c.c. may be made up to 100 c.c. with a freshly prepared solution of sulphurous acid, as mentioned above. After well mixing, the liquid is treated with sulphur dioxide, and after adding a little decolourising carbon if necessary, it is filtered, and polarised at 20° C. in a 200 mm. water-jacketed tube. For the inversion reading, 50 c.c. of the same defecated and filtered normal solution of the molasses as that used for the direct reading are transferred to a 100 c.c. flask, 25 c.c. of water, and 5 c.c. of concentrated hydrochloric acid (sp. gr. 1·19) added, the liquid inverted according to the Herzfeld procedure, and read in the same 200 mm. water-jacketed tube at exactly 20° C. Finally, the percentage of sucrose is calculated from the two readings in the ordinary way, applying, however, the constant from the Herzfeld table appropriate to the particular concentration used. Under these conditions it is unnecessary to take account of the volume of the lead precipitates produced in making the direct and inversion readings; but it is pointed out that it is advisable in operating the inversion polarisation, especially in examining very impure products, to carry out the determination in duplicate, continuing heating at 69° C. in the second test for five minutes longer than in the first. In this way complete hydrolysis of the sucrose in the presence of an abnormally high soluble ash and non-sugar content is insured. J. P. O.

INORGANIC ANALYSIS.

Estimation of Free Acid and Base in Aluminium Sulphate. W. Iwanow. (*J. Russ. Phys. Chem. Ges.*, 1913, **45**, 57-66; through *Chem. Zentralbl.*, 1913, I., 1460-1461.)—The methods generally recommended for the estimation of the free acid or base in aluminium sulphate give inaccurate results. The author, therefore, proposes a method based on the fact that the neutral salts of aluminium are precipitated by potassium ferrocyanide on warming, whilst the acid remains in solution and can be estimated by titration. One grm. of the sample to be tested is dissolved in 50 c.c. of water; the solution is heated to 85° C., and 12 c.c. of a solution of potassium ferrocyanide (1 : 12) are introduced in a rapid stream; then 20 c.c. of a solution of barium chloride (1 : 10) are added. After stirring, the liquid is transferred to a 100 c.c. flask and diluted to the mark; the precipitate is allowed to settle, and 25 to 50 c.c. of the clear solution are withdrawn for titration with $\frac{N}{10}$ sodium hydroxide in presence of methyl orange. If the aluminium sulphate is basic, 2 c.c. of $\frac{N}{2}$ hydrochloric acid are added to the original solution, and this quantity is deducted from the result found in the titration. Test-analyses made have shown that the proposed method affords useful results. For rapid work, the decantation of the clear liquid may be omitted, and the whole may be titrated directly, using as an external indicator methyl orange paper, on which a drop of the solution is placed from time to time during the titration; the results thus obtained, however, are about 0·2 per cent. too low. J. F. B.

Behaviour of Salts of Cæsium and Rubidium towards Salts of Heavy Metals. M. Wagenaar. (*Pharm. Weekblad.*, 1913, **50**, 273-280; through *Chem. Zentralbl.*, 1913, I., 1362-1363.)—When solutions containing cæsium or rubidium salts are mixed with solutions of various heavy metals, characteristic precipitates are obtained in certain cases, and it is shown that a solution of cæsium chloride is a useful reagent for the microchemical detection of antimony, tin, bismuth, copper, cadmium, aluminium, chromium, cobalt, and zinc. Rubidium chloride may be used for the detection of antimony, tin, bismuth, copper, aluminium, and chromium.

W. P. S.

Estimation of Calcium as Tungstate. A. Saint-Sernin. (*Compt. rend.*, 1913, **156**, 1019-1020.)—When excess of a 20 per cent. solution of normal sodium tungstate is added to a boiling ammoniacal solution of calcium chloride, the calcium is quantitatively precipitated as $CaWO_4$. As the corresponding salt of magnesium is easily soluble, the method affords a ready means of separating calcium from magnesium. As the precipitate is crystalline and very heavy (sp. gr. 6·05), it is easily washed by decantation. For the estimation of calcium, it must be collected on a tared filter and dried at 100° C., as burning the paper, even after detaching the precipitate as far as possible, always leads to low results. No test numbers are given. G. C. J.

Analysis of Hydrosulphites. E. Bosshard and W. Grob. (*Chem. Zeit.*, 1913, **37**, 423-425; 437-439.)—After reviewing various methods which have been proposed for the analysis of hydrosulphites, the authors recommend a modification of the process in which the salt is titrated with ammoniacal copper sulphate solution. The procedure is as follows: A weighed quantity of about 0·1 grm. of the salt is placed in an Erlenmeyer flask, and a current of pure hydrogen is passed into the latter. After twenty minutes, and when all the air has been expelled from the flask, ammoniacal copper sulphate solution is run in from a burette, the jet of which passes through the rubber stopper closing the flask. The quantity of copper solution added at first is a few c.c. short of that required to decompose the hydrosulphite completely (that is, to produce a blue coloration in the flask); 2 drops of a 0·1 per cent. indigo-carmine solution are then added, and the titration with the copper solution is continued until the yellow colour of the contents of the flask changes to light blue. The copper solution is prepared by dissolving 4·469 grms. of crystallised copper sulphate in boiled and cooled water, adding a large excess of ammonia, and diluting the whole to 1 litre; the actual quantity of copper per c.c. of this solution must be estimated electrolytically. The reaction proceeds according to the equation:

$$2CuSO_4 + Na_2S_2O_4 + 4NH_3 + 2H_2O = Cu_2SO_4 + Na_2SO_3 + (NH_4)_2SO_3 + (NH_4)_2SO_4.$$

The results found are affected to a slight extent when bisulphites are present in the sample in considerable proportion, the figures obtained being about 0·3 per cent. too high.

Hydrosulphites may also be estimated by an acidimetric method which is based on the reaction:

$$Na_2S_2O_4 + H_2O + O_2 = NaHSO_3 + NaHSO_4.$$

The two hydrogen salts formed are titrated with barium hydroxide solution, using phenolphthaleïn as indicator. The operation is carried out in an atmosphere of hydrogen, and the results obtained agree with those yielded by the copper titration method.

Processes which depend on the reduction of mercuric chloride by hydrosulphites yield fairly accurate results, and may be used for technical purposes. W. P. S.

Monohydrated Ferrous Sulphate and its Employment in Volumetric Analysis. D. Florentin. (*Bull. Soc. Chim.*, 1913, **13**, 362-366.)—Monohydrated ferrous sulphate is recommended as a suitable standard for the control of solutions of permanganate. It is easily prepared, is not hygroscopic, does not oxidise appreciably on storage in a stoppered bottle, and neither loses water nor suffers oxidation when heated for several hours at 120° C. To prepare it, commercial ferrous sulphate is first purified by two or three disturbed crystallisations from very faintly acid solution. Some 400 grms. of the purified salt are then placed in a 500 c.c. flask together with 200 c.c. of 50 per cent. (by weight) sulphuric acid, and heated on a water-bath. The salt begins by dissolving almost completely; then suddenly, on the attainment of a certain temperature, a white crystalline precipitate appears. Heating is continued for a few minutes, and the precipitate is then brought on to a Büchner funnel, washed with 96 per cent. alcohol or anhydrous acetone, then with anhydrous ether, and finally dried in a vacuum desiccator. The yield is about 40 grms. The salt contains 32·93 per cent. of iron (theory for $FeSO_4.H_2O = 32·94$ per cent.), and is free from any but negligible traces of ferric salt. On exposure to the air of the laboratory for fifteen days, 3 grms. appeared to increase in weight 1·5 mgrms., and 4 grms. lost 1·5 mgrms. when kept for four hours at 120° C. G. C. J.

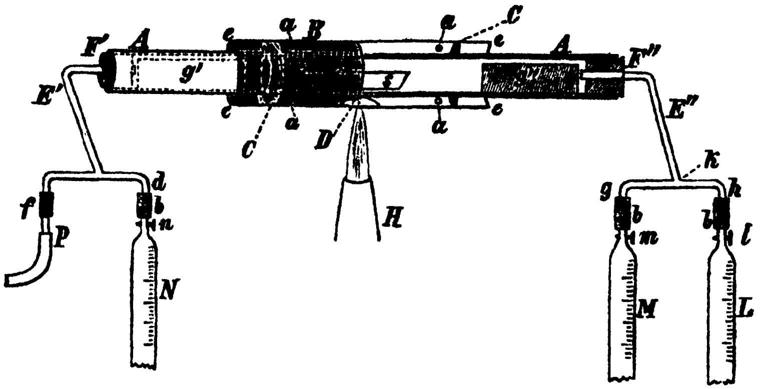
Estimation of Total Carbon in Steel and Ferro-Alloys by Combustion in Oxygen under Pressure. W. E. von John. (*Chem. Zeit.*, 1913, **37**, 426-427.)—An apparatus of simple construction is described for this purpose. The weighed portion of the sample is placed in a magnesia crucible supported in an Erlenmeyer flask by means of a wire attached to a copper tube which passes through the rubber stopper of the flask. Two electrodes also pass through the stopper, and their lower ends extend into the crucible. At the commencement of an estimation the flask is filled with oxygen, a known quantity of $\frac{N}{10}$ sodium hydroxide is introduced, and the rubber stopper, bearing the electrodes and copper tube with the crucible and its contents, is inserted. The upper end of the copper tube is connected with a reservoir of oxygen under slight pressure; a bottle containing mercury is placed between the reservoir and the combustion flask, and serves as a safety-valve. At the end of the combustion, which is carried out under a pressure of about 1 atmosphere, the excess of sodium hydroxide in the flask is titrated, using phenolphthaleïn as indicator, and the quantity of carbon present is calculated from the carbonate formed. The presence of sulphur (up to 0·8 per cent.) and of phosphorus (up to 2 per cent.) in the steel or iron alloy does not affect the accuracy of the method. W. P. S.

Colorimetric Method for Determination of Chromium in Steel. F. Garratt. (*J. Ind. and Eng. Chem.*, 1913, **5**, 298-300.)—The author finds that Koenig's method (ANALYST, 1911, **36**, 238) is better adapted than any other for the estimation of small quantities of chromium in steel from 0·6 per cent. down to the smallest quantity that can have any metallurgical significance. The method is applied to steel as follows: From 0·2 to 0·4 grm. (the larger quantity only for steels with less than 0·15 per cent. chromium) is dissolved in 10 c.c. of dilute (1 : 3) sulphuric acid, 0·5 c.c. nitric acid is added to oxidise the iron, and the solution is evaporated until fumes of sulphuric acid appear. To the cooled and slightly diluted solution 50 c.c. of 10 per cent. sodium hydroxide are added, together with about 1 grm. of sodium peroxide. The mixture is then boiled for five minutes to destroy hydrogen peroxide, cooled, diluted to exactly 200 c.c., and filtered. To 100 c.c. of the filtrate, 2 c.c. of 85 per cent. phosphoric acid, 8 c.c. of concentrated sulphuric acid, and 2 c.c. of Koenig's reagent (a 1 per cent. solution of disodium 1·8-dihydroxynaphthalene 3·6-disulphonate) are added, and the pink colour is compared with that produced by treating an equal weight of chromium-free steel and a measured volume of standard bichromate solution in a similar manner. As about 5 per cent. of the chromium present is carried down with the precipitate of ferric hydroxide, steel must be used in the blank test. Neither tungsten, molybdenum nor titanium interfere. Vanadium interferes by imparting a brownish tint, and where the amount of vanadium equals or exceeds the amount of chromium, the results are worthless. In the commoner case, where the chromium preponderates, the results are too high by about one-third of the vanadium present. Thus a steel with 0·2 per cent. chromium and 0·1 per cent. vanadium appeared to contain 0·23 per cent. chromium when tested as described, and the paper includes a number of analyses showing that the error due to vanadium closely approximates one-third of the vanadium present. When the ratio of chromium to vanadium is high, this error becomes less than the unavoidable error of a colorimetric method, and in other cases it can be reduced to negligible proportions by the use, in the blank test, of a vanadium steel of approximately the same vanadium content as the samples. G. C. J.

Estimation of Manganese in Castings and Steels by the Method of G. von Knorre. M. Huybrechts and N. Joassart. (*Bull. Soc. Chim. de Belg.*, 1913, **27**, 130-137.)—The authors have developed a modification of von Knorre's method for the separation of manganese from large quantities of iron, depending on the decomposition of manganese persulphate by heating, and its precipitation as the dioxide. The authors confirm the observation that the precipitation is very unsatisfactory when any considerable quantity of acid is present, and have taken steps to deal with this difficulty. A complete separation of the iron is not required, but merely a concentration of the manganese in a state in which its estimation may be effected by the Guyard-Volhard or other suitable method. The procedure adopted in the case of manganiferous metals is as follows: Three to four grms. of the metal are dissolved in the least possible excess of nitric acid (11 c.c. of acid of $\frac{N}{7}$ concentration per grm. of sample) in an Erlenmeyer flask, with 40 to 50 c.c. of water, the liquid being heated towards the end of the operation. The solution is diluted with 250 c.c. of water, treated with 50 c.c. of an 8 per cent. solution of ammonium persulphate, boiled for fifteen to twenty minutes, removed from the flame, again treated with 50 c.c. of the persulphate and 50 c.c. of an 8 per cent. solution of sodium carbonate, added together, and again boiled for twenty minutes. The precipitate is filtered off, the filtrate being returned until clear; the precipitate is not washed, but merely drained. A deposit remains on the walls of the empty flask; this is dissolved by running 3 to 4 c.c. of strong hydrochloric acid down the sides of the flask while the latter is still warm. The filter containing the precipitate is placed over the same flask, a hole is made, and the precipitate is washed into the acid by a jet of boiling water. Meanwhile, a mixture of 15 c.c. of $\frac{N}{5}$ hydrochloric acid and 15 c.c. of hydrogen peroxide is prepared and poured, drop by drop, on the filter; in this way the last residues of precipitate are dissolved on the filter and washed into the flask with boiling water. The solution is boiled to destroy the hydrogen peroxide, and is then diluted to a definite volume, and filtered to remove paper fibres. The solution contains all the manganese and a little of the iron; aliquot portions of this are taken for the determination of the manganese by the Guyard-Volhard method. Zinc oxide is added in quantity just sufficient to precipitate the whole of the iron, the addition being stopped when the precipitate of ferric hydroxide is reddish-brown rather than yellowish-brown in colour; an excess of zinc oxide should be avoided. The manganese is titrated at a temperature as near 100° C. as possible. The filtrate from the manganese precipitate should be tested colorimetrically, to ascertain whether any of the manganese has escaped precipitation. It is diluted to 500 c.c., and 50 c.c. are placed in a 100 c.c. flask. To this are added, in succession, 3 grms. of potassium dihydrogen phosphate, 10 c.c. of nitric acid (sp. gr. 1·2) and 5 c.c. of $\frac{N}{10}$ silver nitrate solution. The mixture is heated on the water-bath for ten to fifteen minutes, 3 grms. of ammonium persulphate dissolved in a little water are added, and the solution is shaken, cooled, and diluted to 100 c.c. The colour of the liquid is then compared with standards prepared from permanganate solution. If the acidity of the original solution from which the manganese was precipitated has been sufficiently controlled, this colour test should show no manganese in the filtrate.

J. F. B.

Direct Estimation of Elementary Nitrogen by Means of Commercial Calcium Carbide. B. Natus. (*Zeitsch. anal. Chem.*, 1913, **52**, 265-292.)—The method depends on the fixation of the nitrogen as calcium cyanamide, and its subsequent determination by subjecting the calcium cyanamide to Kjeldahl's process. In the illustration, *AA* is a porcelain tube 30 cm. long by 15 mm. wide; *g′ g″* are 6 cm. lengths of hard glass rod, to reduce the gas-space; *S* is a porcelain boat 5 to 6 cm. long; *B* is an asbestos mantle wrapped round the asbestos rings *CC*. The mantle has a large opening, *D*, for the admission of the hot products of combustion from the burner *H* to the space between the tube and the mantle, and a series of small holes, *aa*, near the end of this space, for the escape of the hot gases, secures a more uniform heating of the boat. The combustion-tube is covered by tiles, not shown in the illustration, and the rubber stoppers *F′ F″* are protected by perforated asbestos discs slipped over the tube, also not shown. *E′ E″* are capillary tubes, and *L*, *M*, *N* are gas burettes, *l*, *m*, *n* being their cocks, and *b*, *b*, *b* rubber connections. *P* connects to a supply of dry hydrogen. The boat is charged with about 2 grms. of a finely powdered mixture of calcium carbide (10 parts) and fused calcium chloride (1 part), ignited at a red heat in a current of dry hydrogen, allowed to cool in the gas,



weighed, and transferred to the apparatus. It is necessary to weigh the carbide mixture, as even after ignition it always retains nitrogen (1 to 2 c.c. per grm.). Each batch of 100 grms. is tested by igniting about 2 grms. as described, and submitting the residue to Kjeldahl's process; a correction is then applied to all results obtained with this mixture. The boat, plugs *g′ g″*, stoppers *F′ F″*, and capillaries *E′ E″* being in place, but the burettes disconnected, *f* is connected to a supply of dry hydrogen, which is passed through the apparatus for fifteen minutes, *d* being plugged after the first few minutes. Meanwhile *L* is charged with the gas mixture, and after fifteen minutes the three burettes, *M* and *N* being full of mercury, are connected, with their cocks closed, to the capillaries. Some 30 c.c. of hydrogen are run over into *M*, the cock *m* again closed, the stream of hydrogen stopped, and *f* plugged. After thirty minutes the volume of gas in *L*, its temperature, and the barometric height are noted, and the combustion-tube raised to 900° to 1,000° C., after opening *n* so that the hydrogen may expand into *N*. A higher temperature than 1,000° C. is to be avoided, lest calcium cyanamide be partially decomposed or sublimed. When the mercury level in *N* no longer sinks, from 12 to 15 c.c. of the gas mixture in *L* are driven into the apparatus, and *n* is closed. The absorption is rapid at first (over 10 c.c. per minute), but gradually becomes slower. If the mixture in *L* is mostly nitrogen, the whole of it can be gradually transferred to the apparatus by manipulating the levelling-tube of *L* so that the pressure is but slightly above that of the atmosphere. Cock *l* is then closed and *m* opened, when, if the gas mixture being examined is mainly nitrogen, as much as 10 c.c. of hydrogen will gradually enter the apparatus to take the place of the absorbed nitrogen. When the level of the mercury in *M* no longer rises, the attached levelling-tube is raised, *n* opened, and the gaseous contents of the apparatus slowly driven over into *N*, and then back into *M*. This operation may be repeated when mercury is used as containing liquid. The apparatus is now allowed to cool, and the boat with its contents, which adhere somewhat firmly to it, is introduced carefully into a Kjeldahl flask, and 25 c.c. of 98 per cent. sulphuric acid added so as to wet the sides of the flask without touching the contents of the boat, which should float. The flask is then agitated so that the contents of the boat are thoroughly and instantaneously wetted. A globule of mercury is added, and the mixture heated to boiling till colourless (one hour); it is then cooled, made alkaline, treated with potassium sulphide, and distilled into standard acid, using zinc dust to prevent bumping. Importance is attached to the use of zinc for this purpose, and to the manner of bringing the material in contact with acid. Different procedure gave erratic results. Working as above, the extreme error (always negative) of the method can be kept below 0·3 c.c. nitrogen. The author's experiments were made with about 50 c.c. of nitrogen.

If water be used as containing liquid, a slight additional correction must be made for nitrogen which may be found in hydrogen, even when this gas is carefully prepared and stored over boiled water; but the chief error introduced by the use of water is due to the partial decomposition of calcium cyanamide by water-vapour. When using water, therefore, the sweeping of the gas back and forth from *M* to *N* should not be repeated more than twice. With this precaution, the error (always negative) can be kept below 0·9 c.c.

G. C. J.

Detection of Nitrous Acid in the Presence of Ferric Salts. P. Altmann. (*Chem. Zeit.*, 1913, **37**, 501.)—Ferric salts do not interfere with iodimetric estimations provided that the solution is nearly saturated with a phosphate, and that an excess of potassium iodide is avoided. Advantage may be taken of this fact for the iodimetric estimation of ammonia in the presence of ferric salts. It is also possible in this way to detect nitrous acid in the presence of ferric salts. Eight grms. of pure disodium hydrogen phosphate are dissolved in 100 c.c. of water containing ferric salts, and the solution shaken with about 0·2 grm. of potassium iodide until only a white turbidity of ferric phosphate is perceptible. The liquid is then acidified with 5 c.c. of $\frac{N}{4}$ sulphuric acid, and 2 c.c. of starch solution added, a blue coloration being at once produced in the presence of a trace of a nitrite. If large quantities of ferric salts are present, the speed of separation of the iodine by the nitrous acid is accelerated. By this test it is possible to detect from 0·1 to 0·2 mgrm. of nitrous acid in the presence of 500 mgrms. of ferric iron per litre. C. A. M.

Precipitation of Palladium by Means of α-nitroso-β-naphthol. W. Schmidt. (*Zeitsch. anorg. Chem.*, 1913, **80**, 335-336.)—When 3 drops of a saturated solution of α-nitroso-β-naphthol in 50 per cent. acetic acid are added to 1 c.c. of a solution containing a trace of palladium, a voluminous red-brown precipitate results. The reaction is shown even by solutions containing only 1 part of palladium in 1,000,000, if some little time be allowed, and none of the other platinum metals give a similar reaction. Applied to the quantitative determination of palladium in pure solutions and in amounts such as 0·02 grm., the results were exact so far as could be shown by a balance sensitive only to 0·1 mgrm. In presence of 1 grm. of platinum and 0·2 grm. rhodium, 0·0190 grm. palladium was returned when 0·0188 grm. was present, so that the method appears suited to the estimation of palladium in presence of much platinum and a considerable amount of rhodium. The solution to be examined is rendered faintly acid with nitric acid, heated to boiling, the reagent added, the precipitate filtered and washed with hot water, the precipitate ignited with the paper in a porcelain crucible, and the residual palladium weighed. It is shown that the precipitate has the composition $(C_{10}H_6NO.O)_2Pd$.

G. C. J.

Detection of White Phosphorus in Match Compositions. F. Schröder. (*Arbeit. Kaiserl. Gesundheitsamte*, 1913, **44**, 1-29.)—The experimental part of this paper is a continuation of the work of A. Siemens (*Arbeit. Kaiserl. Gesundheitsamte*, 1906, **24**, 264). A table of the substances used in the match industry is given; of these, red and scarlet phosphorus, phosphorus sulphides, "sulphophosphite" (zinc polyhypothiophosphite), phosphorus suboxide, and solid hydrogen phosphide are alone likely to contain white phosphorus. The usual tests for phosphorus are not specific for the white variety; the test should, moreover, be insensitive to the small quantities unavoidably left in the commercial product. Siemens (*loc. cit.*) has given a method applicable to red phosphorus, for which the Mitscherlisch test fails. It is also useless for the phosphorus sulphides, except diphosphorus pentasulphide. Experiments on the phosphorescence of the sulphides, pure or contaminated with white phosphorus, led up to the following method: Strips of Schleicher and Schüll No. 597 filter-paper, 3×10 cm., are saturated with 0·5 c.c. of a solution made by dissolving 3 grms. of the phosphorus sulphide, sulphophosphite, red or scarlet phosphorus, or match composition, in 150 c.c. benzene, or, in the case of very sensitive compositions, 200 match heads in 15 c.c. of benzene. The strips are suspended with a thermometer in a vertical glass tube, 50×4·2 cm., and are heated to 45°–50° C. by a current of air rising through the tube from the interior of a closed Victor-Meyer water-bath beneath. The temperature must not in any case exceed 55° C. Screens are arranged to cut off all light from the burner, and the whole apparatus is set up in a dark room. With the quantities given, no phosphorescence should be observed as the benzene evaporates; but if not less than 0·02 mgrm., or, in the case of tetraphosphorus trisulphide, 0·04 mgrm., of white phosphorus is present in 1 c.c. of the benzene solution, the paper becomes distinctly phosphorescent; with more, the intensity is increased, and luminous clouds appear. According to the complete scheme of examination appended, 1 c.c. of the benzene solution is first tested with 1 c.c. of a solution containing 1·7 grms. silver nitrate in 100 c.c. of ammonia of sp. gr. 0·992. If, on shaking and allowing to separate, the aqueous layer is unaltered or tinted yellow, no white phosphorus is present. If it is coloured red or brown, or contains a brown or brown-black precipitate, the rest of the benzene solution must be subjected to the phosphorescence test, unless the substance under examination is red phosphorus, when the presence of white phosphorus is proved and further testing is unnecessary. If required, the presence of phosphorus sulphides is determined by testing the benzene solution with lead nitrate. If the test with ammoniacal silver solution is positive, and the phosphorescence and lead nitrate tests negative, scarlet phosphorus is present.

O. E. M.

Determination of Phosphorus in Ferrotungsten, Metallic Tungsten Powder, Tungsten Oxide, and Tungstic Acid, by Direct Solution. C. M. Johnson. (*J. Ind. and Eng. Chem.*, 1913, **5**, 297-298.)—In the author's experience, the practice of fusing tungsten-bearing materials with sodium carbonate and nitre, leaching out the fusion, removing tungstic acid by evaporation with acid, and using the filtrate from tungstic acid for the determination of phosphorus, leads to the under-estimation of that element. In no case has he succeeded in recovering as much as one-third of the phosphorus present, and in some cases less than one-tenth was recovered, the rest being carried down as phosphotungstic acid with the tungstic acid. The only method he has found sufficiently accurate for technical purposes is the following somewhat lengthy one:

Tungsten ores or tungsten oxide (1 grm.) in the finest possible state of division is extracted for at least six hours at a nearly boiling temperature with 100 c.c. of concentrated hydrochloric acid, adding about 0·1 grm. of potassium chlorate every half-hour or so. When decomposition is complete, the yellow tungstic oxide will show no trace of the brown or grey colour of the original ore. The mixture is then evaporated to dryness, the residue heated for ten minutes with 50 c.c. of hydrochloric acid, 50 c.c. of water are added, the mixture is boiled for fifteen minutes, cooled, and, after addition of some paper pulp, filtered through a double filter. The precipitate is washed with dilute (1 : 20) hydrochloric acid, and the filtrate and washings treated in the manner described in the next paragraph but one.

Tungsten powder is first roasted at a low red heat until converted into the yellow oxide; it is then treated as above described. Ferrotungsten (1 grm.) is decomposed by treatment with 30 c.c. of concentrated nitric acid, with the slow addition of 3 c.c. of hydrofluoric acid, followed by warming, and finally boiling until a clear solution is obtained. A porcelain dish can be used, but a little more hydrofluoric acid must then be taken, and a blank experiment run, using a standard steel, as there is a tendency to get high results when using porcelain. The solution is evaporated to dryness, but not baked; 50 c.c. of hydrochloric acid are added, and the solution is again evaporated to dryness; the residue is heated with 20 c.c. of hydrochloric acid, 50 c.c. of water are added, and the solution filtered from the bulk of the tungstic acid. To separate the last of this, the filtrate and washings, for which dilute (1 : 20) hydrochloric acid is used, are evaporated to 10 c.c., diluted to about 30 c.c., and refiltered.

The filtrate and washings are evaporated to 10 c.c., 75 c.c. of nitric acid are added, and the mixture heated with a cover on until all action is over. It is then evaporated to 20 c.c., 50 c.c. of nitric acid are added, and evaporation is continued until only about 15 c.c. remains. About 20 c.c. of water are then added, the liquid filtered, and the filter washed fifteen to twenty times with 2 per cent. nitric acid. The filtrate and washings are concentrated to 40 c.c., excess of permanganate added, the mixture boiled three to four minutes, manganese dioxide dissolved by means of ferrous sulphate, and the phosphorus precipitated by means of molybdate solution.

G. C. J.

Estimation of Phosphorus in Iron and Steel. P. Artmann and J. Preisinger. (*Zeitsch. angew. Chem.*, 1913, **26**, 203-208.)—Some sixty methods which have been proposed for this estimation are discussed, the opinion expressed that the most accurate of them is probably that which depends on collecting the phosphomolybdate precipitate on a tared Gooch filter, heating to 160° to 180° C. and weighing, and the following method, which is shown to give results in close accord with those obtained by the foregoing method, is recommended on the ground of simplicity. In essentials it is identical with the method of Artmann and Brandis (*Zeitsch. anal. Chem.*, 1910, **49**, 1), and depends on the estimation of the ammonia in the phosphomolybdate precipitate by treating this with a measured excess of standard hypobromite solution and determining the excess of hypobromite iodimetrically. Applied to iron or steel, the method is conducted as follows: The sample (0·3 to 1 grm. cast iron or 2 to 4 grms. steel) is dissolved in 25 to 100 c.c. of nitric acid (sp. gr. 1·135), and after three minutes the solution is evaporated to half bulk. In the case of cast iron, graphite is filtered off and washed with nitric acid (sp. gr. 1·135). With steel 10 c.c. of the acid are added, and then in either case 5 c.c. of 5 per cent. permanganate. The solution is boiled till the permanganate colour disappears, 5 to 10 c.c. of 3 per cent. hydrogen peroxide are added to reduce manganese dioxide, and the solution boiled till clear. The volume of the solution is now increased to 50 to 100 c.c. by addition of nitric acid (sp. gr. 1·135), 5 to 20 grms. ammonium nitrate are added, the mixture heated to boiling, and then withdrawn from the source of heat. To the solution, still at about 95° C., 40 to 60 c.c. of molybdate solution are added slowly, so that the addition occupies about three minutes, the mixture being stirred vigorously the while and for a further five minutes. The molybdate solution contains 75 grms. ammonium molybdate, and 110 c.c. of concentrated nitric acid per litre. The liquid, now at about 40° C., is decanted through a filter, and the precipitate is washed twice by decantation with a 5 per cent. solution of ammonium nitrate containing 0·5 per cent. nitric acid, then transferred to the filter and washed three or four times with the same mixture, and finally five or six times with 8 to 10 c.c. of water. The precipitate and paper are now introduced into a mixture of 10 c.c. standard hypobromite and about 20 c.c. water. The hypobromite solution is about $\frac{N}{5}$, and is made by adding gradually 1,400 c.c. of bromine water to 500 c.c. of well-cooled $\frac{N}{4}$ sodium hydroxide. Sodium phosphate (7 grms.) is next added to inhibit the reduction of molybdic acid by hydrogen iodide, water is added to bring the volume up to about 80 c.c., the mixture agitated to dissolve the phosphate, 0·5 grm. potassium iodide and 10 to 15 c.c. of $\frac{N}{4}$ sulphuric acid are added, and the liberated iodine titrated with $\frac{N}{20}$ thiosulphate. The difference between the volume of thiosulphate required in a blank test and that required in an analysis conducted as described is the measure of the phosphorus present.

$$P = 3NH_3 = 9I.$$

Even proportions of phosphorus as small as 0·02 per cent. can be estimated with an error not exceeding 5 per cent. G. C. J.

Separation of Phosphoric Acid by Means of Stannic Acid in Qualitative Analysis. W. Mecklenburg. (*Zeitsch. anal. Chem.*, 1913, **52**, 293-298.)— The author has recently shown (*Zeitsch. anorg. Chem.*, 1912, **74**, 215) that Reynoso's method for the separation of phosphoric acid in the course of qualitative analysis, by boiling the nitric acid solution with tin, depends on the adsorption of phosphoric acid by metastannic acid. Some skill is required to use the original method successfully, but the substitution of ready-made hydrated stannic oxide for tin makes the separation quite easy. The reagent is prepared by adding gradually 150 grms. of granulated tin to 1,500 c.c. of ice-cold dilute nitric acid (sp. gr. 1·2). The colloidal sol is poured into 10 litres of water at room temperature, and the resulting precipitate is washed several times by decantation, filtered off on linen, washed till the washings are nearly neutral, and allowed to drain. The gel on the filter, which contains about 10 per cent. of stannic oxide, constitutes the reagent. As much as 40 grms. is required to adsorb 0·1 grm. of phosphoric acid at one operation, but the reagent is more efficient if used as described below. The solution to be analysed, freed from metals of Groups I. and II., and containing about 15 c.c. of nitric acid in 100 c.c., is treated with a tablespoonful of the gel, boiled for fifteen minutes, and filtered. The filtrate is treated in a similar manner, the first runnings of the second filtrate rejected if cloudy, and the subsequent clear filtrate collected. In it any metals except iron, titanium, and zirconium, which were present in the original solution, can be detected. With as much as 0·7 grm. phosphoric acid present in the original solution, less than 1 mgrm. finds its way into the filtrate. If the original solution contains chlorides or large quantities of iron, aluminium, or chromium, some tin may pass into solution. It may be separated, together with any copper or lead derived from the original "tin," by means of hydrogen sulphide, before proceeding with the analysis, but in absence of chlorides the necessity for this is rare. Zirconium was not experimented with, but it is pointed out that it forms a compound with phosphoric acid which is insoluble, even in strong nitric acid. Iron and titanium are wholly retained by the gel, and may be tested for by simple treatment of the gel with ferrocyanide and hydrogen peroxide respectively. If traces of these metals have to be looked for, it is best to make the first separation with very little of the gel and to filter this off and test it. This concentrates the iron and titanium into a small bulk, and enables very small quantities to be detected.

G. C. J.

Iodimetric Estimation of Persulphuric Acid. E. Müller. (*Zeitsch. anal. Chem.*, 1913, **52**, 299-303.)—When a persulphate is added to excess of a solution of sodium hydroxide and potassium iodide, the solution first acquires a yellow colour due to hypoiodite, but finally becomes colourless, and then contains iodate. The reaction, which may be represented by the equation—

$$3K_2S_2O_8 + KI + 6KOH = 6K_2SO_4 + KIO_3 + 3H_2O,$$

proceeds slowly at room temperature, but is complete in less than three minutes at 100° C. As it proceeds quantitatively, it affords a ready means of estimating persulphates, the solution of iodate with excess of iodide being acidified and the resulting iodine titrated with thiosulphate. It will be seen that the net result of the reaction between persulphate and iodide, and the well-known one between iodic and hydriodic acids, is that $K_2S_2O_8 = I_2$, or that 0·5 grm. of potassium persulphate will give a convenient reading (about 40 c.c.) if $\frac{N}{10}$ thiosulphate be employed. The process is conducted as follows: to 25 c.c. of $\frac{N}{2}$ sodium hydroxide contained in a flask with 2 grms. potassium iodide, about 0·5 grm. of the salt to be analysed, or an equivalent quantity of a solution, is added and the mixture boiled gently for three minutes. It is then cooled, acidified by addition of 30 c.c. $\frac{N}{2}$ sulphuric acid, and titrated with thiosulphate. Sodium hydroxide purified by alcohol is to be avoided, as it usually exercises a reducing action on persulphate.

If an acid solution is to be analysed, it may be necessary to take a larger quantity of sodium hydroxide than is specified above. The necessity will be shown by the immediate separation of iodine, but it does not suffice merely to decolourise this by addition of alkali; there must be a fair excess of the latter present as the first reaction uses up alkali. The method is unaffected by Caro's acid, which is always present in solutions of persulphuric acid which have been prepared some days. On the other hand, it breaks down in presence of hydrogen peroxide, which is also liable to be present in such solutions, so that its use is practically limited to the analysis of solid substances and neutral solutions.

G. C. J.

Estimation of Platinum. L. St. Rainer. (*Oesterr. Zeit. Berg. u. Hüttenwesen*, 1913, **61**, 141-147, 155-160; through *Chem. Zentralbl.*, 1913, I., 1542-1543.)—For the estimation of platinum in alloys, a representative sample of the repeatedly-fused metal, and (0·25 grm. to 1 grm. in the case of poor alloys) is weighed with sufficient refined gold and silver to give a ratio of Pt : Au : Ag = 1 : 10 : 30. At the same time a control test is made with an equal weight of refined silver, which is cupelled simultaneously with the alloy mixture and with an equal quantity of lead flux. The cupellation is started at 900° C., and the heat afterwards raised to 1,200° C. until the operation is finished, after which the cupels are left for ten minutes in the opening of the muffle furnace and then cooled to increase the malleability of the buttons. The latter are now weighed and the amount of added gold and silver deducted from the platinum button, the quantity of silver being checked by the result obtained in the control cupellation. Both buttons are then rolled in the usual way, and boiled for thirty minutes with sulphuric acid (sp. gr. 1·84), after which the flask is cooled and the boiling repeated for thirty minutes with fresh acid. The residue is washed with dilute sulphuric acid and with water, and then digested for several minutes with nitric acid, followed by an excess of ammonia, or with an ammoniacal solution of ammonium acetate, to dissolve any lead sulphate present, after which the rolls, now consisting of platinum and gold, are dried, ignited, and weighed, and the amount of added gold deducted. Each roll is then alloyed with three times its weight of refined silver, and the resulting buttons again made into rolls, boiled with nitric acid, and subsequently weighed. This treatment is repeated twice to separate the small amounts of platinum retained by the gold. Provided the proportions given of silver are present, the whole of the platinum will be dissolved after the second treatment. A similar method may be used in the examination of platinum dross, but in that case the control cupellation of silver may be omitted.

C. A. M.

Cobaltinitrite Method for the Estimation of Potassium. J. L. M. van der Horn van den Bos. (*Chem. Weekblad*, 1913, **10**, 182-186; through *Chem. Zentralbl.*, 1913, I., 1362.)—The following procedure is recommended: The solution under examination is evaporated, and the residue is treated with 3 c.c. of a 50 per cent. cobaltous chloride solution and 3 c.c. of a 50 per cent. sodium nitrite solution, the mixture is stirred thoroughly, then acidified with 2 c.c. of 50 per cent. acetic acid, and placed aside for forty-eight hours. The precipitate is collected on a filter, washed, and then rinsed into a beaker, an excess of $\frac{N}{50}$ potassium permanganate is added, the mixture warmed, and, after the addition of 15 c.c. of sulphuric acid (1 : 1), the excess of permanganate is titrated with oxalic acid solution. The precipitate has the formula $K_2NaCo(NO_2)_6$, and according to theory 1 c.c. of $\frac{N}{50}$ permanganate should be equivalent to 0·000156 grm. of K_2O, but experiment has shown that 1 c.c. of $\frac{N}{50}$ permanganate corresponds with 0·000172 grm. of K_2O.

W. P. S.

New Physico-Chemical Volumetric Method. R. Dubrisay. (*Compt. rend.*, 1913, **156**, 894-898.)—It was found by Donnan that the superficial tension at the surface of separation of water and a petroleum hydrocarbon was very greatly reduced by the presence of a trace of alkali in the water, when the hydrocarbon contained a fatty acid, such as stearic acid, in solution. On this observation the author has based a method of determining the exact point of neutralisation of strong acids. The liquid under examination is introduced into a curved pipette which is dipped into the water, upon which there is vaseline oil previously purified and saturated with stearic acid. The point of the pipette is made to coincide with the juncture of the oil and water, and the number of drops issuing from the pipette into the oil during the fall of the liquid between two fixed points is then counted. Thus, for example, the following results were obtained with hydrochloric acid 20 c.c. of which required for neutralisation 18·8 c.c. of $\frac{N}{20}$ sodium hydroxide solution:

Number of c.c. of alkali added to 20 c.c. of acid	0	17·9	18·6	18·7	19·1	19·7
Number of drops	45	45	46	45·5	75	98

Similar results were obtained with nitric and sulphuric acids. In the case of boric acid the hydrolysis of the alkali salt was clearly indicated, whilst phosphoric acid at first behaved like a strong acid, but soon showed signs of the hydrolysis of the disodium salt, and subsequently of the neutral salt. C. A. M.

Detection of Potassium by Means of Tartaric Acid. L. W. Winkler. (*Zeitsch. angew. Chem.*, 1913, **26**, 208.)—As is well known, this test is apt to fail owing to the tendency of potassium bitartrate to form supersaturated solutions. The superior efficacy of the powdered reagent, as compared with a solution, is due to the presence of a trace of potassium salt, seldom absent from commercial samples of tartaric acid. The author recommends the following modification of the test: About 0·5 grm. of sodium acetate is first dissolved in 10 c.c. of the solution to be examined, or in 10 c.c. of a 5 per cent. solution of a solid, and 0·5 grm. of powdered commercial tartaric acid is then added, and the mixture shaken. In absence of potassium (ammonium, rubidium, and caesium), the solution becomes clear at once, as the powdered acid dissolves rapidly, but 20 mgrms. of potassium will give an immediate precipitate, and 10 mgrms. will give one within two minutes. The reaction succeeds equally well with halides, nitrate, chlorate, and sulphate, and with salts of organic acids except tartar emetic. Potash alums react somewhat less well. The use of alcohol is apt to make the test less certain, as it may throw out other salts than potassium bitartrate. G. C. J.

Preparation of Selenic Acid and Sodium Selenate as Reagents for the Estimation of Bromine in Halogen Salts. P. L. Blumenthal. (*Zeitsch. anorg. Chem.*, 1913, **80**, 246-251.)—Gooch and Blumenthal have described a method (Analyst, 1913, 227) for the estimation of bromide in presence of chloride, which depends on the use of selenic acid or sodium selenate. When these reagents are prepared by the usual methods (Gmelin-Kraut, *Handb. d. anorg. Chem.*, 1907, I., 769) they are apt to retain impurities which interfere with their use for the above purpose. Selenic acid of a satisfactory degree of purity may be prepared as follows: Selenious acid is first prepared by dissolving elementary selenium in nitric acid and evaporating to dryness. The crude product is sublimed over manganese dioxide (*cf.* Norton, *Zeitsch. anorg. Chem.*, 1899, **20**, 222), and 10 grms. of the sublimate are treated with 75 to 100 c.c. water, 6 grms. potassium bromate, and 4 c.c. nitric acid, and the mixture is heated on the water-bath. Bromine is set free, and selenic acid results, and on evaporating the solution to small bulk, the excess of bromic acid decomposes the nitric acid with liberation of more bromine. When this reaction is over and the solution is colourless, more bromate is added, little by little, so long as an addition gives rise to the liberation of bromine, showing that some nitric acid is yet undecomposed. When the solution remains colourless on addition of more bromate, it is made faintly alkaline with ammonia, heated to boiling, and the selenic acid precipitated by means of barium nitrate. The barium selenate is filtered off at the pump, washed with hot water, and dried at a red heat. It is then decomposed by means of 5 c.c. of sulphuric acid, 20 c.c. of water is added, and the mixture heated for two to three hours on the water-bath, diluted, filtered, and washed until the filtrate and washings amount to about 30 c.c. The resulting solution is a 40 per cent. solution of selenic acid, containing about 5 per cent. of selenious acid, but free from any impurity which would interfere with its use in the estimation of bromine.

Sodium selenate is prepared by mixing 105 grms. of powdered selenium with 500 grms. of sodium peroxide, and fusing the mixture in batches of about 50 grms. in nickel crucibles. The fusion only occupies a few minutes. The cooled melt is dissolved out with water, and the solution filtered through asbestos, and evaporated as rapidly as possible over a free flame to a syrupy consistency. The bulk of the caustic alkali is then extracted by means of hot 97 to 99 per cent. alcohol, and the extraction repeated six or seven times. The residue of sodium selenate and carbonate is dissolved in water, and the solution carefully neutralised by means of sulphuric acid, and evaporated to incipient crystallisation, and allowed to cool. The crystals are separated from the mother liquor, which is further evaporated to obtain a fresh crop. The several crops of crystals are united and dried at 108° C. They consist of about 70 per cent. sodium selenate and 30 per cent. sodium sulphate. The first crop contains more sulphate, and a certain degree of fractionation is possible, but is not worth while, as sodium sulphate is not an objectionable impurity, and any attempt to eliminate it reduces the recovery of selenium, which in no case exceeds 60 per cent. G. C. J.

Determination of the Specific Gravity of Mineral Powders. M. Billy. (*Compt. rend.*, 1913, **156**, 1065-1067.)—To prevent the inaccuracies caused by the air retained between the particles of a mineral powder, the water used in the determination of the sp. gr. may be replaced by a solution of potassium hydroxide (of about $\frac{N}{1}$ strength), and the air by carbon dioxide. Any bubble of gas will then no longer increase the volume of the powder, but will be absorbed by the alkali solution. A special form of sp. gr. bottle for this purpose is described, in which air is replaced by carbon dioxide at the same temperature and pressure. The sp. gr. of the alkaline solution is taken at 4° C., under the same conditions as that of the powder.

C. A. M.

New Process for Titration of Sulphurous Acid and of Sulphites in Presence of Thiosulphates. E. Bosshard and W. Grob. (*Chem. Zeit.*, 1913, **37**, 465-466.)—When excess of mercuric chloride is added to a solution of a bisulphite, the solution, previously neutral to methyl orange, reacts acid towards that indicator, and continues to do so until sufficient alkali is added to convert the whole of the bisulphite into normal sulphite. The end-point is much sharper than that obtained in the usual titration with phenolphthaleïn as indicator, and the results are exact if some sodium chloride is added to counteract the tendency of the mercuric chloride to become partially hydrolysed and react acid. In mixtures of normal and acid sulphite, the former is first titrated with hydrochloric acid, using methyl orange as indicator, and the resulting solution of bisulphite is retitrated with caustic alkali, after addition of 1 grm. of sodium chloride and 300 c.c. of a saturated solution of mercuric chloride. The above observation of the effect of a large excess of mercuric chloride on the behaviour of bisulphites towards indicators shows that Feld's method for the estimation of thiosulphates (*Zeitsch. angew. Chem.*, 1911, **24**, 290; ANALYST, 1911, **36**, 435) would fail in accuracy in presence of sulphites or bisulphites. That method depends on the decomposition of thiosulphates, in accordance with the equation: $Na_2S_2O_3 + 2HgCl_2 + H_2O = Na_2SO_4 + Hg_2Cl_2 + 2HCl + S$. Mixtures of normal and acid sulphite with thiosulphate may be analysed as follows: The normal sulphite is titrated with hydrochloric acid, using methyl orange as indicator (1), and the resulting solution of bisulphite is re-titrated with caustic alkali in presence of phenolphthaleïn (2). A further portion of the solution is then treated with a large excess of mercuric chloride and 1 grm. of sodium chloride, and, after addition of 30 c.c. of 20 per cent. ammonium chloride (to prevent precipitation of mercuric oxide), is titrated with caustic alkali, using methyl orange as indicator. From the amount of alkali used, that due to the bisulphite originally present [calculated from the difference between titrations (2) and (1)] is deducted, and the remainder calculated to thiosulphate in accordance with Feld's equation. G. C. J.

Detection of Tin. L. J. Curtman and M. Mosher. (*J. Amer. Chem. Soc.*, 1913, **35**, 357-365.)—A study of the limits of sensitiveness of the mercuric chloride and ammonium molybdate tests and of the conditions which determine the maximum sensitiveness, starting with a stannic solution containing antimony such as is separated in the course of a qualitative analysis. Under the most favourable conditions the mercuric chloride test will detect 0·2 mgrm. of tin in 5 c.c. The most favourable conditions are reduction of the tin in $\frac{N}{5}$ hydrochloric acid solution by heating 5 c.c. with one 1½-inch cut iron nail for three minutes on the water-bath, and filtering into a tube containing 2 c.c. of saturated mercuric chloride solution. As much as 250 mgrms. of antimony are without influence on the result.

The molybdate test is even more sensitive than the above test if carried out exactly as follows: Five c.c. of the stannic solution, which should contain about 1·5 c.c. of concentrated hydrochloric acid, are heated on the water-bath with not more than 0·5 grm. of granulated zinc. When action has practically ceased, the precipitated tin is dissolved by addition of a further 1 c.c. of hydrochloric acid, and the heating continued. The solution is then diluted to 10 c.c., and filtered into 1 c.c.

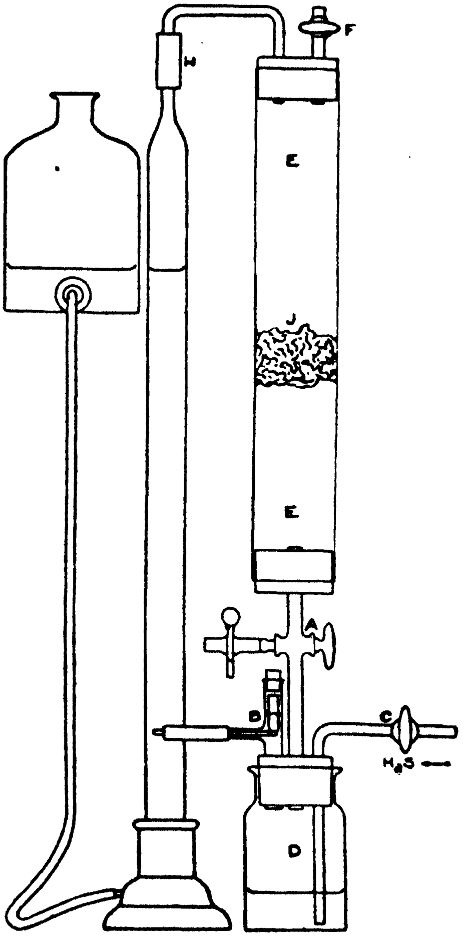
of saturated ammonium molybdate solution. Under these conditions, and in absence of antimony, as little as 0·01 mgrm. of tin will give a faint but distinct blue coloration. The presence of 250 mgrms. of antimony reduces the limit of sensitiveness of the test to 0·05 mgrm. tin; with 10 mgrms. of antimony present, 0·03 mgrm. tin can be detected, whilst 5 mgrms. of antimony are without measurable effect. The sensitiveness of this test is diminished by a higher concentration of acid than that above given. For example, if the final solution contains 5 c.c. of concentrated acid in 10 c.c., even as much as 1 mgrm. of tin may be overlooked. G. C. J.

Determination of Zinc in Ores. D. J. Demorest. (*J. Ind. and Eng. Chem.*, 1913, **5**, 302-304.)—The following modification of Low's method is applicable to any ore except Gahnite (zinc spinel), which must first be fused with sodium carbonate and borax glass. One grm. of the ore is heated for several minutes with 5 c.c. of hydrochloric acid, 20 c.c. of nitric acid are added, and heating is continued with the cover on until brown fumes cease to be evolved. One grm. of potassium chlorate is then added, and the mixture is evaporated to dryness. About 50 c.c. of water are added together with not more than 0·5 grm. of potassium hydroxide, and the cake is broken up. The purpose of the potassium hydroxide is to neutralise any trace of nitric acid, which otherwise would subsequently form ammonium nitrate, in which cadmium carbonate is somewhat soluble. About 6 grms. of ammonium carbonate are added, and the solution is kept near to the boiling-point for several minutes. The precipitate, which contains all the iron, aluminium, manganese, lead, and cadmium, is filtered off, preferably by suction, and washed with hot 5 per cent. ammonium carbonate. It is then washed back into the beaker, dissolved in 3 c.c. of hydrochloric acid, a little potassium nitrite is added to reduce manganese dioxide, and the solution is neutralised by means of caustic potash, of which no excess should be used. Ammonium carbonate (5 grms.) is again added, and the mixture heated to boiling for several minutes, and filtered again through the original filter. Very small precipitates need not be redissolved and reprecipitated. The filtrate, which should not exceed 200 c.c., is neutralised with hydrochloric acid, of which an excess of 20 c.c. (sp. gr. 1·19) is then added. After heating to 70° C., hydrogen sulphide is passed through the solution, which precipitates most of the copper without any co-precipitation of zinc owing to the high concentration of acid. When no further precipitation occurs, 20 c.c. of dilute (1 : 1) ammonia (nearly but not quite enough to neutralise the acid present) are added slowly with constant stirring, and the stream of hydrogen sulphide is continued for several minutes. This completes the precipitation of the copper. Then, without filtering or expelling hydrogen sulphide, the solution is heated again to 70° C. and titrated with ferrocyanide, using uranyl nitrate as indicator. Five tests of an ore, issued by the U.S. Bureau of Standards as containing 31·41 per cent. of zinc, gave results varying from 31·30 to 31·45 per cent. Addition of salts of cadmium, copper, and lead to the original solution had no influence on the results. G. C. J.

ERRATUM.—On page 81 of the Journal (February, 1913), line 17, *for* D. J. Davenport, *read* D. J. Demorest.

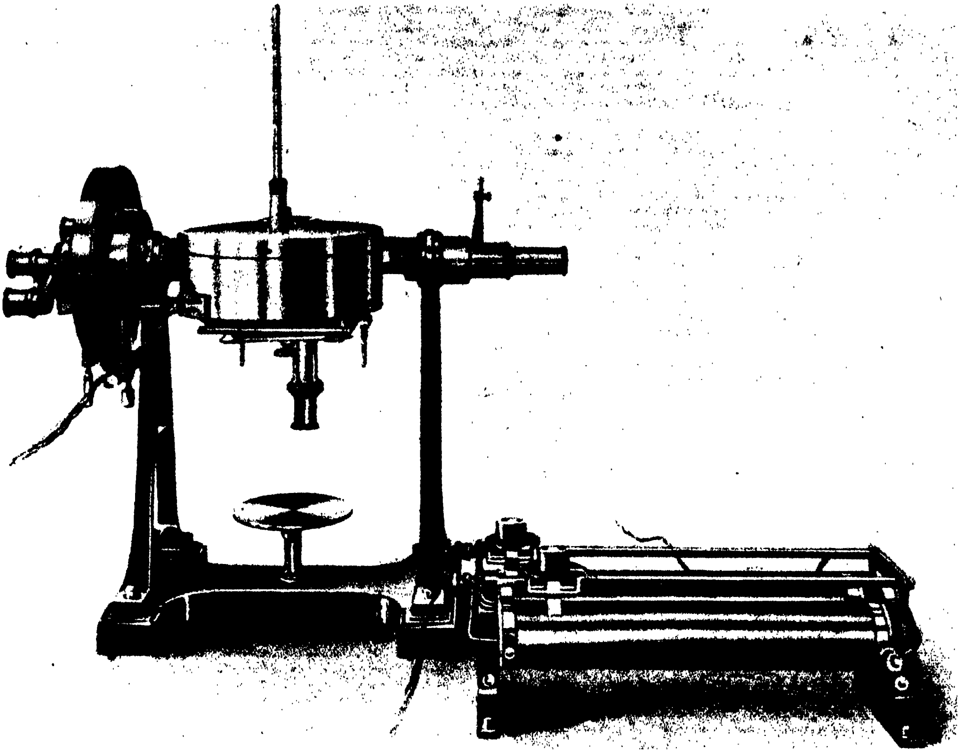
APPARATUS, ETC.

Improved Apparatus for Determination of Carboxyl Groups in Organic Acids. W. H. Hunter and J. D. Edwards. (*J. Amer. Chem. Soc.*, 1913, **35**, 452-461.)—The improvement relates to Fuchs' method for the estimation of carboxyl groups (*Monatsh.*, 1888, **9**, 1132, 1143; 1890, **11**, 363) by measurement of the hydrogen sulphide which is evolved when even weak organic acids are brought into contact with a solution of potassium hydrosulphide saturated with hydrogen sulphide. The method has the advantage over the titration method that most lactones and alcohols, and phenols as such, are without effect, but with the apparatus hitherto in use, only roughly approximate results could be obtained. The possible sources of error are discussed, and an apparatus described which eliminates all but two, which appear unavoidable, and to which reference is made later.



In the illustration *A* is a three-way cock, such that the vessels *D* and *E* can be connected together, or either of them can be opened to the atmosphere through the pinchcock shown at the left. *B* is the sample tube, supported as in some forms of Victor Meyer vapour density apparatus, *D* is a bottle 50 mm. in diameter, and 80 mm. high to the shoulder, *E* is a glass tube about 37 mm. in diameter, and 360 mm. from stopper to stopper, and containing near its middle a loose plug of cotton-wool. The apparatus on the left is a Hempel burette with levelling cistern. The apparatus is set up as shown, but without the sample. The cock *F* is connected to a drying apparatus and dry air drawn through *E* for five minutes by applying suction at the three-way cock. Then 25 c.c. of 10 per cent. caustic potash, freed from carbonate by treatment with a slight excess of baryta and filtering, are introduced into *D*, the cock *A* is turned to connect *D* with the atmosphere, and hydrogen sulphide is allowed to enter through the cock *C* until all the air is expelled. The pinchcock on the side limb of cock *B* is now closed, as the neutral point on this type of cock is not trustworthy, the sample holder is opened to let out air, the sample is introduced (in the position shown, not allowed to fall into the solution) and the stopper replaced. When gas ceases to bubble into the solution, *D* is held by the upper edge and shaken for five minutes. The pinchcock is now removed and the solution shaken three minutes with the gas flowing through. The stream of gas is then slowed down and the apparatus given thirty minutes to cool to room temperature. Hydrogen sulphide derived from commercial ferrous sulphide may contain enough hydrogen to vitiate the results. The authors generate the gas in the ordinary way, absorb the hydrogen sulphide in strong potash, and use the resulting solution for generating hydrogen sulphide of sufficient purity. The gas burette levelled at about 99 c.c. is connected with the air chamber and levelled again, the hydrogen sulphide is cut off, and connection made between *D* and *E*. The gas burette is again levelled, and *D* shaken for two minutes. No change in level greater than 0·1 c.c. should result. The reading of the burette is then taken, and the sample allowed to fall into the hydrosulphide solution. As gas is evolved, the levelling cistern is lowered to keep the pressure in the apparatus constant, and, when no more gas is evolved, a reading is taken, *D* is shaken for three minutes, and a further reading taken. A small increase of volume usually, but not always, results. The volume of wet gas is then reduced to dry gas at N. T. P., and calculated to - COOH. In the authors' hands the individual results of experiments performed in triplicate never differ by more than 0·2 per cent. from the mean. Monobasic acids—such as crotonic, anisic, benzoic, and salicylic acids—give results between 97·6 and 97·9 per cent. of the theoretical; whilst dibasic acids—such as maleic and tartaric acids—give results which are about 100·7 per cent. of the theoretical. Theoretical considerations are advanced to show that results of this kind might be expected; that the higher results with maleic and tartaric acids are dependent, not on the greater strength of those acids, but on the fact that they are dibasic; and that with dibasic acids the negative error due to partition of the potassium kation between the hydrosulphide anion and the anion of the acid under investigation is halved, and thus ceases to outweigh the unavoidable (positive) error due to the calculation of the gas to dry hydrogen sulphide, whereas it is really saturated with water at an unknown pressure—namely, that of water vapour over 10 per cent. potassium hydrosulphide. G. C. J.

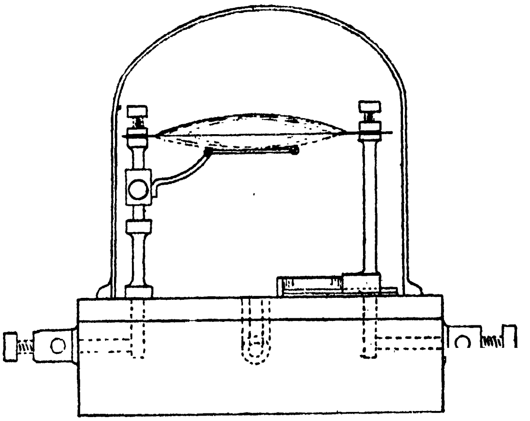
Electrically Heated Apparatus for Measuring Rotatory Power at Constant Temperature. E. Abderhalden. (*Zeitsch. Physiol. Chem.*, 1913, **84**, 300-305.)—The author found that water-jacketed tubes do not afford sufficient protection against cooling for inexperienced workers, and therefore constructed an apparatus, in collaboration with the firm Schmidt and Haensch of Berlin, by means of which the tubes can be maintained at any desired temperature. The apparatus, which can be attached to any large polarimeter as in the figure, consists of an electrically heated metal vessel the temperature of which can be regulated and maintained with precision by adjusting a resistance. This vessel is provided with a tightly fitting lid having one opening for thermometer, and a second opening, fitted with a separate lid, for introducing and removing the observation tubes without taking off the large lid. These tubes are so arranged that they can be rotated round an axis and thus brought successively into the field of vision. The tubes are automatically secured, so that the axis of the tube coincides exactly with that of the polarimeter. The apparatus is constructed to take six tubes of 2·5 cm. length and 2 c.c. content, but can be modified to take longer and more tubes. The tubes are numbered, and the apparatus is provided with an index with corresponding numbers, so that when the apparatus is charged it is possible to observe the rotation of any desired tube without disturbing the heated chamber. The author has used the apparatus for fermentation experiments at 37°, and in this connection warns against a possible source of error arising from the formation of an optically active sediment which may sink and escape observation, thus causing a change of rotation without the occurrence of ferment action. To guard against this the tubes should be rapidly removed after observation, inverted, and again observed, when any precipitation will become apparent.



E. W.

Modification of the Liebig Combustion Method. H. Brach. (*Zeitsch. angew. Chem.*, 1913, **26**, 230.)—The apparatus consists of two concentric combustion-tubes, each packed in the usual manner, and heated in an electric furnace. To enable the combustion to be completed in thirty minutes without loss, the gases, after passing through the weighed calcium chloride tube attached to the inner combustion-tube, and giving up all their water, are led through a weighed soda-lime tube, where most of the carbon dioxide is absorbed, and thence through the space between the combustion-tubes. The remainder of the carbon dioxide is absorbed in a second weighed soda-lime tube, to which is attached a washbottle containing palladium chloride solution, in order that any escape of carbon monoxide may be detected, and the rate of flow ot the gases regulated. Details of the precautions necessary are given. O. E. M.

Apophorometer. J. Joly. (*Chem. News*, 1913, **107**, 146-149.)—Under this name the author describes a piece of apparatus for collecting sublimates and determining their weight, etc. The form of the apparatus is shown in the figure. A ribbon of thin platinum, about 6 cms. in length and 5 mm. in width, is stretched between two forceps, which are provided with binding screws, so that an electric current can be passed through the ribbon, raising its temperature to any desired degree up to the melting-point of platinum. One of the forceps is movable, and is so acted on by a light spring that the ribbon is kept stretched. Beneath the ribbon a watch-glass is held in position by a support, which can be raised or lowered, or rotated to one side. From 5 to 30 mgrms. of the substance are placed on the ribbon and distributed over nearly its entire length; a second watch-glass is placed over the first, and a receiver is fitted over all, so that the experiment may be carried out *in vacuo* or in an atmosphere consisting of an indifferent gas, as required. The temperature of the ribbon is raised gradually, until the substance, or one of its constituents, begins to sublime, and the temperature is then maintained unaltered until the sublimation is completed. In certain cases, more than one sublimate may be obtained by careful regulation of the temperature. Before the watch-glasses have become cold after the current has been cut off, they are removed, clipped together, and weighed. In some instances the platinum ribbon may be weighed before being placed in position. Moulded strips of carbon may be substituted for the platinum ribbon in dealing with substances which would alloy with the platinum and cause it to fuse. A very large number of minerals may be analysed partially by the aid of this apparatus, and much useful information obtained as to their composition, etc. Numerous applications of the process to minerals are given. W. P. S.



Quantitative Manipulation of Small Precipitates. J. Donau. (*Monatsh.*, 1913, **34**, 553-560.)—The apparatus previously described by the author for conducting precipitations (ANALYST, 1912, **37**, 68) serves well enough when the bulk of the precipitate is not too small, but, with very small quantities of crystalline precipitates, the quantity adhering to the precipitation vessel may correspond to an error considerably in excess of 0·5 per cent. Apparatus is now described by means of which the error may be reduced to a few tenths of 1 per cent., even when the quantity being manipulated is as small as 1 mgrm. The apparatus consists of a basin of platinum, provided with a handle of foil or thin wire for grasping by means of forceps. The basin is weighed together with the filter pan previously described (*loc. cit.*). The substance for analysis is added, its weight determined by difference, dissolved and precipitated in the basin, the precipitate filtered and washed, and the filter and basin again brought to the balance together after drying. The precipitating basin is about 22 mm. in diameter and provided with a handle of foil about 1·5 to 2 cms. long and 1 mm. broad. It is desirable that it should be as light as possible, but it must be free from pinholes, and the minimum weight consistent with freedom from pinholes is attained by constructing it from two, or if necessary three, discs of very thin foil (0·0025 mm. thick) welded together. Directions for cutting and welding the discs, shaping them into basins, and using them are given in the paper. G. C. J.

Notes on Thermometry. J. H. Coste. (*J. Soc. Chem. Ind.*, 1913, **32**, 341.) —The errors of mercury thermometers are reviewed, and information on the composition and physical properties of the glasses used in their construction is given. Different types and forms of thermometers are compared, and precautions in their use and standardisation, with several useful definite temperature points, are given, together with some limits on the employment of copper-constantan thermocouples. O. E. M.

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REVIEWS.

THE LABORATORY BOOK OF DAIRY ANALYSIS. By H. DROOP RICHMOND, F.I.C. London: Charles Griffin and Co., Ltd. 1912. Second edition. Pp. 100. Price 2s. 6d. net.

This little work is intended to assist analysts, health officers, dairy students, and those engaged in the supervision of dairies. In it the author has endeavoured to describe the more simple tests in such a manner as to enable them to be carried out by persons devoid of chemical training, and to help such as these he gives large photographic illustrations of himself and his assistants in the act of carrying out some of the more simple analytical tests. Four illustrations are devoted to the use of the lactometer, showing this instrument being put into and being taken out of milk. There are also pictures of the most simple laboratory apparatus and of people using pipettes, weighing cream, and washing precipitates. To one who has never seen any analytical operations or apparatus these pictures should prove most helpful; but the analyst who casually opens the book and is confronted with them might disdainfully leave it for his less scientific brethren. However, in so doing he would make a mistake, for in addition to the elementary matter he will find concise yet sufficiently detailed descriptions of practically every valuable test commonly applied to dairy products. Even the experienced dairy chemist could not read this book without finding many of those valuable hints which can only be given by one who has had long experience in the processes he describes, and who, at the same time, shows himself to be alive to the pitfalls likely to trap the beginner.

The author must be congratulated upon the amount of readily accessible information contained in so small a compass and upon having produced a book so useful to the novice, but which also contains such information, data, and tables as will render it a valuable help to the more experienced worker. E. R. Bolton.

Organometallic Compounds of Zinc and Magnesium (Chemical Monographs, No. 1). By Henry Wren. London: Gurney and Jackson. 1913. Pp. 100 + viii. Price 1s. 6d.

The basis of all analytical methods is the chemical reactions on which the processes of determination depend, and a knowledge of pure chemistry is becoming more and more important to the analyst. Though this work deals hardly at all with analytical matters, it does deal with a very important series of reactions, which may form the basis of future analytical methods.

As the preface states, "the progress of chemistry is so rapid that it is becoming a matter of ever-increasing difficulty to keep abreast of the modern developments of the science," and a summary such as this is almost a necessity for those who have not ample time to search through the vast volume of periodical literature.

This work is divided into four sections, which treat respectively of the Griquard reaction, of the products formed by the aid of Grignard's reagents, of the theory of the subject, and of zinc organometallic compounds; and there is a complete table of references. The whole subject is covered in a manner which not only allows the student to grasp easily the facts, but provides, in addition, a guide to the investigator.

H. Droop Richmond.

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THE INSTITUTE OF METALS.

GHENT MEETING.

The Annual Autumn Meeting of the Institute of Metals will this year be held at Ghent, under the presidency of Professor A. K. Huntington, A.R.S.M. It will take place in connection with the Ghent International Exhibition, the dates fixed being August 28, 29, and 30. Among many important papers to be communicated will be the Report of the Corrosion Committee.

Those who desire to attend the Meeting should send in their forms of application for membership to the Secretary of the Institute of Metals, Caxton House, Westminster, S.W., at the earliest possible moment, and not later than July 31.

Elliott & Fry, photographers, London Emery Walker sc.

JULY, 1913. Vol. XXXVIII., No. 448.

THE ANALYST.

OBITUARY.

M. A. ADAMS, F.R.C.S., F.I.C.

It falls once more to the lot of the writer to give expression to his great sorrow at the loss of a dear friend and colleague, a prominent member and past President of the Society of Public Analysts. Matthew Algernon Adams passed away, 76 years of age, on April 29, at Bearsted, Kent.

Adams was born in 1836 in London. He studied medicine at Guy's Hospital, hearing chemistry lectures by Odling and Taylor. After having been house-surgeon to the Public Dispensary, in Leeds he settled, soon after 1860, as a medical practitioner in Maidstone. He specialised in ophthalmic surgery, in which he excelled and earned an almost international repute. Hard-working and well-read, he had wide scientific interests, with a predilection for chemistry. In 1874, while the Sale of Food and Drugs Bill was under Parliamentary discussion, Adams worked for some time in the laboratory of J. A. Wanklyn, and was in the same year appointed Public Analyst for the county of Kent. He held that position till 1911, when he was succeeded by his former assistant, then partner, L. W. Stansell. He also was analyst for Rochester and Maidstone.

He was one of the medical men who, when the Food and Drugs Act compelled local authorities to appoint public analysts, developed in an iatro-analytical era into chemists. A number of them—like Letheby, Hassall, Tidy, Wanklyn, Stevenson, Hill, Adams, and Cameron, became distinguished both as sanitarians and as analysts. At that time the exercise of the small amount of knowledge available for food analysis could easily be added as an appanage to a medical practice. Delicate questions involving an accurate knowledge of the chemistry of food had not yet arisen, and there was fair reason why appointing authorities should entrust to the doctors, who to them were the apostles of all that was scientific, the duty of controlling the food-supply. Chemistry and the processes carried on in laboratories are even now, after a generation, impenetrable mysteries to local administrators. At that time, as now, the doctor, to the popular mind, represented the knowledge of nature, from medicine to astronomy, from geology to sewage-treatment, from sanitation to adulteration. Many of the medico-chemists—Adams among them—rendered splendid service, and the now independent profession of chemistry owes them a deep debt of gratitude. In Germany the modern chemist developed from the apothecary; in England, upon a less chemical but broader humanitarian medical basis.

Adams for many years regularly attended the meetings of the Society. His capacity for clear exposition at the meetings, his sound common sense in the Council, his geniality and kindness in the Chair, greatly endeared him to the members.

An observation made by him in 1884 that cooked (not raw) apple-pulp contained a substance reacting with iodine like starch, enabling the ready distinction of apple in mixtures with other fruits, has been found exceedingly useful in the microscopical examination of jams.

About 1880, and for some years afterwards, an amount of controversy, which now seems hardly intelligible, raged round the composition and analysis of milk. Wanklyn had, some years before, stated upon somewhat slender data that milk, apart from a considerable variation in the fat percentage, was of remarkably constant composition, the amount of "solids-not-fat" ranging from 9·0 to 9·3 per cent. While this statement had never met with acceptance by Continental chemists, public analysts had adopted Wanklyn's methods of milk analysis and his figures almost without criticism. The method adopted by Wanklyn for the separation of fatty and non-fatty constituents was inconceivably crude, and consisted in the direct extraction of the hard, horn-like, and impervious masses of casein and sugar, obtained by the evaporation of milk in small basins with a few successively applied portions of ether. Of course, anyone now can see that the whole of the fat could not thus be extracted. Workers like Vieth, then at the Aylesbury Dairy Company, trained in other schools, had always insisted upon a subdivision of the solids by gypsum or other powdered mineral matter. But no one had realised how widely the discrepancies of analysis of the same sample of milk could be in various, otherwise competent, hands, until a celebrated milk case, heard in Manchester in 1885, caused the appointment by the Society of a Milk Committee to investigate the matter. Portions of the same milk were distributed and analysed by the members of the Committee, each one at first following his own laboratory method. The discrepancies were relatively enormous. While various proposed modifications were being investigated, Adams's mechanical genius solved the question in the simplest possible manner. By merely distributing a weighed quantity of milk over strips of blotting-paper and thus subdividing the solids over a very large surface, the direct extraction of the fat became easy and complete. It is true that Adams at first overlooked the fact that all blotting-paper contains substances which, partly under the influence of the action of the milk, become soluble in ether and thus add to the apparent percentage of fat—an error at once detected and corrected by Vieth. But we owe to Adams the simple solution of a problem which at that time was of urgent importance, and it can truly be said that from the introduction of the Adams method accurate milk-analysis takes its date, and that it at once swept away all previously published results. It has become the standard method by which all other methods of fat-estimation in milk are judged and regulated. The observation in itself was a small one, but it has affected each one of the fifty thousand milk analyses annually made by English and Scottish public analysts.

Living in the midst of the Kentish hop industry, Adams was greatly interested in the chemistry of the hop. He worked out a process for preserving new hops, and preventing them from undergoing deteriorating changes which vastly affect their price. He also devised a process for the distinction of hop-bitter from quassia and other bitter substances in malt beverages.

He was President of the Society of Public Analysts in 1889 and 1890, and a Fellow of the Institute of Chemistry, and of other chemical and scientific bodies.

To his activity as Medical Officer of Health and Sanitarian no detailed reference need be made in the ANALYST. His services to the Borough of Maidstone in the suppression of diphtheria and smallpox, and at the time of the terrible outbreak of typhoid fever in 1897, secure him the lasting gratitude of his fellow-citizens.

For many years I was in intimate and very frequent contact with him in London and at his home in Bearsted, where, in the middle of lovely Kentish scenery, he had built himself a house on a hill affording a wide panoramic outlook. The kindest memories will ever connect him with many of my colleagues and myself, who had deplored that as a result of a serious illness some nine years ago he had been compelled to forego attendance at our meetings. OTTO HEHNER.

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PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

THE monthly meeting of the Society was held on Wednesday evening, June 4, in the Chemical Society's Rooms, Burlington House. The President, Mr. L. Archbutt, F.I.C., occupied the chair.

The minutes of the previous ordinary meeting were read and confirmed.

Certificates of proposal for election to membership in favour of Messrs. U. R. Evans, B.A., C. W. Birch, and H. W. Gill, B.Sc., A.I.C., were read for the second time; and certificates in favour of Messrs. John Joseph Eastick, A.R.S.M., F.I.C., analytical and consulting chemist, 2, St. Dunstan's Hill, London, E.C., and J. F. Millar, chemist to the Cawnpore Sugar Company, Ltd., Cawnpore, India, were read for the first time.

Mr. F. S. Sinnatt was elected a member of the Society.

The following papers were read: "An Electro-chemical Indicator for Oxidisers," by Eric K. Rideal, B.A., and Ulick R. Evans, B.A.; "The Estimation of Tannin in Tea," by Henry L. Smith, B.Sc., F.I.C.; "The Detection and Estimation of Nickel by means of α-Benzildioxime," by F. W. Atack, M.Sc.; "The Analysis of Various East-Indian Tanned Hides," by M. C. Lamb; "A General Method for the Detection of Caramel," by P. F. Thompson; and a "Note on the Sterilisation of Rag Flock Samples," by Lester Reed, F.I.C.

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CONSTANT TEMPERATURE APPARATUS.

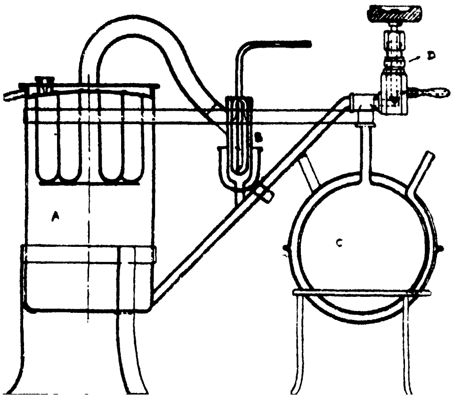
By F. H. and P. V. DUPRÉ.

(*Read at the Meeting, May* 7, 1913.)

Having for some years past had occasion to carry out work which necessitated a very accurate regulation of temperature, in some cases for many hours at a time, and not having been able to find anything on the market which was entirely satisfactory, we have designed an apparatus which has proved extremely efficient.

The object we set before ourselves was to design something that would give a wide range of temperatures, with a very high degree of accuracy, for any length of time required, and not requiring supervision. We also wished, if possible, to avoid the use of an inflammable liquid, and to obtain this large range of temperatures without any modification of the apparatus or alteration in the liquid with which it was filled. This object we think we may fairly claim to have attained.

In the first place, we will describe the construction of the apparatus and the principle of its action; secondly, a description of our method of using it; and, thirdly, what we consider to be its advantages and the degree of accuracy obtainable.



The apparatus, which is based on the principle that the temperature of the steam given off by water, boiling under constant pressure, is constant, consists of a boiler, which is about one-third full of water, a reflux condenser, a water-jacketed pressure bulb, and a screw valve, marked *A*, *B*, *C*, *D* respectively in the figure. When the screw valve is shut, the whole apparatus is absolutely air-tight. A fusible metal safety plug is supplied.

When the boiler is heated by a gas flame, or by any other suitable source of heat, the steam generated drives all the air from the boiler over into the pressure bulb, thus causing a definite pressure in the latter, depending on the total amount of air present originally in the whole apparatus, the steam being condensed and returned to the boiler. This pressure determines the temperature of the steam evolved in the boiler, and so long as sufficient heat is supplied to keep the water boiling without producing more steam than can be dealt with by the condenser, the temperature remains practically constant. Within these limits the amount of heat supplied to the boiler has very little effect on the temperature, and as the type of condenser which we use can deal easily with the amount of steam produced in a two-gallon still by a gas burner consuming 20 cubic feet of gas per hour, it is obvious that a very large variation in the size of the gas flame employed is permissible.* As the pressure in the bulb will obviously be affected by changes of temperature, the water supplied to the condenser is first passed through the jacket of the pressure bulb. As it is necessary for the temperature of this water to remain constant, it should be taken either from a large cistern, or, preferably, from the main direct.

Method of Use.—The exact temperature required can readily be obtained as follows:

When temperatures of above 100° C. are required, the screw valve is opened so as to fill the apparatus with air at atmospheric pressure. The valve is then closed, the cooling water turned on, and the gas lighted. After a short time all the air in the boiler is driven over into the bulb, the relative volumes of boiler and bulb being so arranged that this will give the maximum pressure, and consequently the maximum temperature, for which the apparatus is constructed. If this temperature is higher than that required, the valve is cautiously opened and air allowed to escape until the desired temperature is reached. The valve is then screwed down tightly, when the temperature will remain constant so long as the amount of heat supplied to the boiler is within the limits laid down above. Moreover, the apparatus may be allowed to cool, and provided that the valve is not opened, and the temperature of the cooling water is unchanged, on applying heat again, the same temperature will be reached and maintained.

If temperatures below 100° C. are required, the apparatus is exhausted to such a degree that a temperature lower than that which is actually required will be produced. The valve is then cautiously opened and air allowed to enter the apparatus until the required temperature is attained. The valve is then tightly closed, when the same conditions as previously described will obtain.

If an ordinary high-pressure, water-jet pump is employed and the apparatus exhausted when cold, a temperature of about 35° C. will be obtained. If lower temperatures than this are required, the apparatus can be further exhausted when the water is boiling, care being taken to have the water boiling as gently as possible so as to avoid any steam being sucked out of the apparatus; in this way a temperature of about 27° C. will be obtained.

We have made a number of experiments and find that, without taking any precautions to screen the apparatus from draughts, etc., or to regulate the gas-supply, the maximum variation will be less than ±0·05° C. however long the apparatus is going, or at whatever the temperature at which one is working, always provided that the temperature of the cooling water is unaltered. At temperatures of

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[*] As an experiment the amount of gas supplied to the burner was measured: it was found to be at the rate of 9·7 cubic feet per hour, the temperature of the apparatus being 71° C. The gas was turned down, the rate being then measured, and found to be 3·7 cubic feet per hour; the temperature then fell to 70·85° C.

100° C. a variation of 1° C. in the temperature of the cooling water makes a difference of about 0·1° C. in the temperature of the apparatus ; at lower temperatures variations in the temperature of the cooling water make rather less difference than this, at higher temperatures rather more. With a regulated gas-supply, and with cooling water direct from the main, we have been able to keep the temperature constant for about an hour at a time, within +0·001° C. We believe it would be quite possible, by suitable modifications, to insure a variation of not more than this for longer periods.

With the apparatus we have had made any temperature between 25° and 145° C. can be maintained. Higher temperatures than these can be obtained by making the apparatus stronger ; lower temperatures we have not experimented with.*

Advantages.—(1) The avoidance of any inflammable liquid, or of one which might change its boiling-point, owing to decomposition caused by prolonged heating. (2) The attainment of the whole range of temperatures with one apparatus, containing only one liquid, and with no other manipulations than those mentioned above. (3) Extreme regularity of temperature. (4) Automatic regulation of temperature, no supervision whatever being required when once the apparatus is properly going. (5) The temperature at which the apparatus has been set is reached very quickly. The water can be turned on, the gas lighted, and the bath left with the knowledge that at any time after, say, fifteen minutes a constant temperature will be reached. As the apparatus cannot possibly go higher than it is set for, there is no need to watch it while the temperature is rising. (6) If the bath has been going continually, and the temperature is the same at the beginning and at the end of any period, however long, it is certain that the temperature has remained constant during the whole of that period within the limits mentioned above. For example, if the temperature is right when leaving it in the evening, and is still right the next morning, there is no fear that the temperature may have varied during the interval. (7) There is no need of a stirring device. (8) As a comparatively high temperature may be employed, the apparatus is but little affected by draughts, temporary cooling of any part, etc. (9) When a cold body is introduced into the apparatus, its temperature is very quickly raised owing to the large amount of heat given up by the condensed steam. (10) The apparatus cannot get out of order while out of use. (11) Constant temperatures are maintained for any length of time. (12) Alterations of room temperature or of barometric pressure have no influence on the apparatus. (13) No regulation of the heating flame is required unless extreme accuracy is necessary. (14) The temperature can be changed, and a new constant temperature obtained in a very short time.

We have also devised a small piece of apparatus to facilitate the adjustment of the temperature. It is simply a means of estimating the quantity of air entering or leaving the apparatus, by causing it to pass through water, enabling the bubbles of air to be counted. The adjustment is a very delicate one, as a large number of bubbles are required to alter the temperature by 1° C.

We are indebted to Messrs. Baird and Tatlock, of London, for assistance in the construction of this apparatus.

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* The exact form of the apparatus depends on the purpose for which it is required ; the form shown in the figure is intended for heat test work.

DISCUSSION.

Mr. HEHNER remarked that the arrangement, as shown, the design of which was most ingenious, appeared to be limited in its utility to the heating of small tubes, such as test-tubes.

Mr. F. H. DUPRÉ said that, although the apparatus shown was made only for heating test-tubes, the boiler might be made spherical, with a compartment which could be opened, and within certain limits its size could be varied considerably. The advantage over the ordinary water-oven was that a temperature of, say, 130° C. could be maintained just as well as 100° C. could be in an ordinary water-oven that was boiling hard. A cylindrical water-oven of a reasonable size could also be made.

Mr. W. T. BURGESS asked if the authors could give a little more information as to how the readings would be affected by any variation that might occur in the initial temperature of the cooling water, upon the constancy of which the efficiency of the apparatus must depend.

Mr. DUPRÉ said that it was certainly necessary that the temperature at which the cooling water was admitted should be constant, and it was therefore desirable to draw it either from a very large cistern or, as they did, from the main. In tests which they had made, extending over periods of about three weeks, they had found the temperature of the water drawn from the main to vary only to the extent of about a hundredth of a degree during the day.

Dr. STEVENS said that if the boiler could be made in the form of a jacketed pan, which could be kept at 130° to 140° C., it would probably be much more useful. It would seem, however, to be easier to maintain a constant temperature in a boiler of the shape shown than when the boiler formed the jacket of a pan, since in the latter case the space to be kept at a constant temperature would be much larger. The variation in the ordinary jacketed pan would not be more than 0·5° C. either way with a gas regulator.

Mr. DUPRÉ said that the difficulty in the case of a jacketed pan would be due to the pressure. At 145° C. the pressure would be between three and four atmospheres, while at 35° C. it would be only about an inch, and in either case the apparatus would have to be very strong in order to avoid leakage at the joints. If the pan could be satisfactorily constructed, the temperature could probably be kept constant within 0·5° C. A spherical vessel of reasonable size would, of course, be easy to make.

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THE DETERMINATION OF TANNIN IN TEA.

By HENRY L. SMITH, B.Sc., F.I.C.

INVESTIGATION CARRIED OUT UNDER THE SOCIETY'S ANALYTICAL INVESTIGATION SCHEME.

(*Read at the Meeting, June* 4, 1913.)

CONSIDERABLE doubt has been thrown on the accuracy of the permanganate process of estimating tannins, as it depends on the assumption that only tannins are precipitated by the saline gelatin solution.

The nature of the tannin in tea is not known with any certainty, and different permanganate equivalents have been used by various workers. Results are given sometimes in terms of gallotannic acid and sometimes in terms of oxalic acid, the latter being stated to have very nearly the same equivalent as quercitannic acid. Tea tannin resembles quercitannic acid rather than gallotannic acid in the readiness with which insoluble anhydrides are formed.

The work described in this paper is an attempt to apply the cinchonine method as devised by A. C. Chapman (*J. Inst. Brewing*, 1907, **13**, 646, and 1909, **15**, 360) for the estimation of tannin in hops to the estimation of the tannin in tea. The method employed is substantially the same as that described by Chapman. It depends on the complete precipitation of tannin by means of a saturated solution of cinchonine sulphate, the cinchonine tannate being weighed.

Caffeine is also precipitated to some extent by tannins, and it was found necessary to remove this alkaloid before precipitating with cinchonine. If the caffeine is not removed, some is carried down with the cinchonine tannate, and cannot be removed by washing with more cinchonine sulphate solution, nor by digestion of the dried precipitate with chloroform. The filtrate from the cinchonine tannate always gave a colour and precipitate on the addition of ferric salts, but after washing with a half-saturated solution of cinchonine sulphate, further washings gave no such colour. This colour was not the typical bluish-black obtained with gallic acid, but was somewhat greenish. To ascertain what substance remained in solution which gave the colour, tea infusions were precipitated with lead acetate, and the precipitate collected and washed. It was then suspended in water and decomposed by means of sulphuretted hydrogen and filtered. The filtrate was boiled to remove sulphuretted hydrogen, concentrated, and dialysed. But little tannin passes the dialyser. The liquid outside the dialyser was evaporated to dryness and extracted with dry ether. The residue after evaporating the ether was very small. From several experiments insufficient material was obtained to purify in order to determine the melting-point. Under the microscope crystals were seen that closely resembled gallic acid, crystallised from ether. A solution of the substance in water gave no precipitate with gelatin nor with cinchonine sulphate. With ferric chloride a bluish-black colour was obtained.

Other tea infusions were treated with gelatin and filtered. The filtrate was concentrated and treated with a large volume of alcohol and again filtered. The alcohol was then distilled off and the residual liquid filtered. Lead acetate was then added, the lead compound collected and decomposed in the usual way with sulphuretted hydrogen. The filtrate from the lead sulphide was then evaporated to dryness and extracted with dry ether. The residue, after distilling off the ether, was evidently not a pure substance, and attempts at purification failed. The whole dissolved readily in water and in alcohol, and somewhat less readily in ether; benzene dissolved nothing. Under the microscope crystals could be seen that differed from gallic acid and from the crystalline residue previously mentioned. Ferric salts gave a greenish colour, which changed to violet and then red on the addition of sodium bicarbonate. It reduced ammoniacal silver nitrate, but not Fehling's solution. With lead acetate it gave a light buff precipitate, but with gelatin and with cinchonine sulphate it gave no precipitate. The substance showed signs of melting at about 197° C., but did not completely melt at 220° C. In its chemical behaviour it resembled protocatechuic acid or some derivative of it.

A very small quantity of a substance giving similar reactions was obtained from the filtrates from a number of quantitative experiments with cinchonine sulphate. Barium carbonate was added to the filtrate and the precipitated cinchonine and barium sulphate filtered off. The filtrate was concentrated to small bulk, made just acid with sulphuric acid and extracted with ether. On distilling off the ether the substance was left. Nothing resembling tannin, and giving a precipitate either with gelatin or with cinchonine sulphate was found in these filtrates.

Cinchonine sulphate, therefore, appears to quantitatively precipitate the tannin of tea under proper conditions. The substances not precipitated, and which give colours with ferric chloride, are not tannins.

Determination of Nitrogen in the Precipitated Cinchonine Tannate.—The nitrogen in a number of preparations of cinchonine tannate from tea was determined by combustion with copper oxide. In some cases the substance was collected from several quantitative experiments. In others the substance was prepared on a larger scale, and, after washing, was pressed on a porous plate and finally dried at 100° C. to constant weight.

The following results were obtained:

Tea.	Weight of Substance. Grm.	Percentage of Nitrogen.
Indian A ...	0·3856 ...	4·45
Indian B ...	0·4143 ...	4·44
	0·2979 ...	4·40
Indian C ...	0·2964 ...	4·29
	0·6602 ...	4·29
Ceylon	0·3975 ...	4·32
	0·4353 ...	4·31
China A ...	0·3806 ...	4·33
	0·6573 ...	4·37
China B ...	0·6197 ...	4·28
China C ...	0·6907 ...	4·22

The average percentage of nitrogen is 4·32 in the cinchonine tannate. The figure 4·3 has been used in calculating the tannin in tea. This is also the figure adopted by Chapman in his analyses of cinchonine lupulo-tannate. The variations in the percentage of nitrogen may be due either to the presence of tannins of different molecular weight, or to the formation of anhydrides.

Attempts were made to extract the tannin and prepare cinchonine tannate from it. Different results were obtained according to the method employed for extraction. The lead compound obtained by precipitating tea infusions with lead acetate was decomposed by means of sulphuretted hydrogen. The filtrate was heated to remove sulphuretted hydrogen and filtered when cold. Two methods were then employed: (1) The liquid was extracted with ethyl acetate and the ethyl acetate removed by distillation under reduced pressure. The residue was yellowish-brown, and dissolved readily in water. Cinchonine tannate prepared from this gave the following results:

0·5199 grm. gave 21·0 c.c. N at 11° C. and 758 mm. N = 4·75 per cent.
0·4585 grm. gave 18·6 c.c. N at 6·5° C. and 748 mm. N = 4·74 per cent.

(2) After boiling off the sulphuretted hydrogen the solution was dialysed for twelve hours, then concentrated and filtered.

The cinchonine tannate from this gave the following results:

0·3015 grm. gave 10·6 c.c. N at 15° C. and 761 mm. N = 4·08 per cent.
0·7021 grm. gave 24·7 c.c. N at 13° C. and 747 mm. N = 4·04 per cent.

In preparing both these specimens of tannin a considerable amount of precipitate formed, which was removed by filtration. On keeping a solution a day anhydrides were precipitated. From these results alone it is not possible to state that different tannins exist in tea, as it is very possible that the methods of extraction caused changes in the nature of the tannin.

Estimation of Tannin by Means of Cinchonine Sulphate.—The decoctions were prepared by boiling 10 grms. of the tea in 800 c.c. water for half an hour, filtering hot, and washing the leaves with 200 c.c. boiling water. When cold, the volume was adjusted to 1,000 c.c. Digestion at 100° C., and allowing the liquid to cool in contact with the leaves before filtering, gave low results. The decoction, on cooling, is always somewhat turbid, especially in teas of high tannin content. But little actual precipitation occurs, they can, however, be filtered bright. This removes some tannin, as the particles causing the turbidity contain both tannin and caffeine. Fifty c.c. of the cold decoction were treated with four successive amounts of 30 c.c. of chloroform, which was found to be sufficient to remove practically all the caffeine. The liquid became clearer on removal of the caffeine. The liquid was then evaporated to about one-third its bulk, and 50 c.c. of a saturated solution of cinchonine sulphate added while still hot and clear. It was allowed to stand some hours, and then filtered through asbestos in a Gooch crucible. The prepared crucible was previously washed with a half-saturated solution of cinchonine sulphate and dried at 100° C. The precipitate was also washed with half-saturated cinchonine sulphate solution and thoroughly drained with suction. It was found advisable to dry the precipitate over sulphuric acid first, and to save time a vacuum desiccator was used; finally it was dried to constant weight at 100° C. If the damp precipitate was dried in the oven it fused, and it was difficult to get constant results, but if first partially dried over sulphuric acid fusion did not occur. Consistent results can be obtained by this method, as shown in the analyses of tannin in a few typical teas in the table.

The cinchonine precipitate is assumed to contain 4·3 per cent. N, and as cinchonine contains 9·52 per cent. N, the precipitate will contain 45·1 per cent. cinchonine and 54·9 tannin, provided that it is anhydrous. The precipitate has been assumed to contain 55 per cent. of tannin, as the process is not of such accuracy as to warrant the use of the figure 54·9.

In some cases the tannin has also been estimated by the permanganate method, Procter's modification of Loewenthal's process being employed. The results are calculated as oxalic acid.

After detannating the decoction with gelatin and the acid salt solution, the filtrate gave no colour with ferric chloride; but on carefully neutralising, a greenish-black colour was obtained.

It will be noticed that the figures in terms of oxalic acid are always low. Whether this is due to the permanganate equivalent of the tannin being incorrect or some other reason, it is not possible to decide in the present state of our knowledge of the tannins of tea.

Tea.	Moisture.	Precipitate from 50 c.c. Decoction. Grms.	Percentage of Tannin by Cinchonine.	Percentage by $KMnO_4$ as Oxalic Acid.
Indian A ...	—	0·137 0·138 0·137 0·136	15·1	13·9
Indian B ...	6·60	0·154 0·155	16·9	15·8
Indian C ...	6·25	0·150 0·150	16·5	—
Ceylon	7·95	0·132 0·132	14·5	12·4
China A ...	8·15	0·104 0·106 0·106	11·6	9·1
China B ...	10·10	0·122 0·123 0·123	13·5	
China C ...	—	0·110 0·110	12·0	9·0

Discussion.

Mr. A. Chaston Chapman said that in the case of the tannin of hops precipitation with quinine was not nearly so satisfactory as precipitation with cinchonine, the cinchonine precipitate being more insoluble and the results more concordant. He was interested to see that the cinchonine precipitate obtained with tea tannin was so similar in composition to that yielded by hop tannin, because in some respects these two tannins appeared to differ rather widely. He also had found, in the case of hop tannin, that something always came through the filter which gave a coloration with ferric chloride, but he had demonstrated that this was not tannin. As to the determination of nitrogen, while cinchonine itself yielded all its nitrogen readily by the Kjeldahl process, the results in the case of this cinchonine tannate were always low; and even by the Dumas process it was difficult to get all the nitrogen unless cuprous chloride were used. The difficulty of determining tannin lay not so much in the actual estimation of the tannin, as in getting it into solution without decomposition. He should have expected Indian tea, judging from its flavour, to contain more tannin than Mr. Smith had found to be the case.

Mr. Chaston Chapman said that the object of his work on the determination of tannin in hops had been to ascertain whether the proportion of tannin was really of any practical consequence. It used to be supposed that the higher the proportion of tannin the greater was the value of the hops for brewing purposes, and that the tannin diminished on storage; but on investigation it was found that there was no connection between the proportion of tannin and the brewing value of the hops, and that the tannin did not diminish, but remained practically constant. He had made a few experiments on the application of the method to tannin materials, and the results appeared to be satisfactory, but he had not gone very far in that direction.

Mr. E. R. Bolton asked whether Mr. Smith had determined the theine in any of these samples. It had been suggested that the ratio of tannin to theine was an important factor in the valuation of tea.

Mr. Smith agreed that the ratio of caffeine to tannin was more important than the actual percentage of tannin. He had not applied the method to any other material than tea, with the exception of one experiment made with coffee, which was negative in value.

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THE DETECTION AND ESTIMATION OF NICKEL BY MEANS OF α-BENZILDIOXIME.

By FREDERICK WILLIAM ATACK, M.Sc. Tech., B.Sc., A.I.C.

(*Read at the Meeting, June* 4, 1913.)

The use of dimethylglyoxime as a reagent for nickel was first suggested by Tschugaeff (*Ber.*, 1905, **38**, 2520). The reagent was applied by Kraut (*Zeitsch. angew. Chem.*, 1906, **19**, 1793) for the gravimetric determination of small amounts of nickel. Its general application for the gravimetric estimation of nickel is due to O. Brunck (*Zeitsch. angew. Chem.*, 1907, **20**, 834 and 1844), who gave details of the estimation of nickel in presence of cobalt, iron, manganese, chromium, and zinc, and later (*Stahl u. Eisen*, 1908, **28**, 331) applied the reagent to the determination of nickel in nickel steel and in chromium nickel steel. The method of weighing the nickel dioxime precipitate on filter-papers was used by A. Iwanicki (*ibid.*, 1908, **28**, 1546; 1909, **29**, 1154). H. Wdowiszewski (*ibid.*, 1908, **28**, 960; 1909, **29**, 358) and F. Ibbotson (*Chem. News*, 1911, **104**, 224) have suggested the ignition of the nickel precipitate. In his "Report on Gravimetric Methods for the Determination of Nickel in Nickel Steel" (Analyst, 1910, **35**, 97), E. L. Rhead found the dimethylglyoxime method to be the most convenient one available for the estimation of nickel in presence of iron. La. V. W. Spring (ANALYST, 1911, **36**, 524) recommends the dimethylglyoxime reagent for the determination of nickel in nickel-zinc alloys and in German silver.

A new reagent for nickel is now proposed which has decided advantages over the dimethylglyoxime reagent—viz., α-benzildioxime. This reagent is available for the detection and estimation of nickel, giving an intense red precipitate which consists of the nickel salt, $C_{28}H_{22}N_4O_4Ni$, one hydrogen atom in each of two molecules of the dioxime being replaced by metal. This compound was described by Tschugaeff (*Zeitsch. anorg. Chem.*, 1905, **46**, 144), but the reaction does not appear to have been applied as a qualitative test or for the gravimetric determination of nickel.

QUALITATIVE APPLICATION OF THE REAGENT.

An alcoholic solution of α-benzildioxime has been found to constitute a very delicate test for the presence of nickel, being an even more characteristic reagent for nickel than the dimethylglyoxime reagent suggested by Tschugaeff. For qualitative purposes a 0·02 per cent. solution of the reagent is dissolved in alcohol to which 5 per cent. of dilute ammonia solution has been added. This solution is quite stable, and has been kept for several months. The reagent can also be used in acetone solution. In either case the solution to be tested is made ammoniacal before addition of the reagent. The new reagent appears to have several distinct advantages over the dimethylglyoxime reagent. Several series of comparative tests have led to the following conclusions:

Nickel only Present.—It was found possible to detect immediately less than 1 part of nickel in 2,000,000 parts of water, using a volume of 5 c.c., corresponding to 0·002 mgrm. of nickel, by means of α-benzildioxime, which gave a copious precipitate, whereas it was necessary to allow a corresponding test with dimethylglyoxime to stand twenty-four hours before the coloration appeared. In general it may be remarked that the new reagent invariably gives an almost immediate reaction if sufficient nickel is present. The rapidity with which the reaction occurs with α-benzildioxime admits of the reagent being applied conveniently as a ring-test. The results with both dimethylglyoxime and α-benzildioxime (α-diphenylglyoxime) are seriously affected by the presence of large amounts of nitrates.

In Presence of Cobalt.—Whereas freshly prepared ammoniacal cobalt solutions (free from nickel) containing 1 part of cobalt in 50,000 parts of water give an intense reddish-violet coloration and pinkish meniscus with dimethylglyoxime, the same solution was found to give only a faint yellow coloration with the α-benzildioxime reagent. In presence of 100 times the amount of cobalt the direct detection of nickel in ammoniacal solution of concentration 1 of nickel in 1,000,000 of water is easy by means of the new reagent, a copious precipitate being obtained almost immediately from a total volume of 10 c.c. The reaction is much more characteristic than with dimethylglyoxime, with which no precipitate was obtained after standing three days, although the colour was then certainly different, though not distinctively so, from a blank containing the same amount of cobalt. The solution effect of the presence of cobalt on the nickel dimethylglyoxime reaction was mentioned in Brunck's original paper. Larger amounts of cobalt must be oxidised by shaking in ammoniacal solution before carrying out the test.

In Presence of Iron.—Slavik (*Chem. Zeit.*, 1910, **34**, 648) has shown that ferrous salts give an intense pink colour with dimethylglyoxime in ammoniacal tartaric or, preferably, citric acid solution—a method which the present writer has found very satisfactory for the rapid detection of small amounts of iron in presence of titanous and similar salts. Hence it is necessary to oxidise ferrous salts before testing for nickel with the dimethylglyoxime reagent. If occasion should arise for the detection of nickel in presence of ferrous salts, the α-benzildioxime reagent may be applied, as the violet coloration produced with ferrous salts does not mask the nickel reaction.

In presence of ferric salts the reagent has been applied with success for the direct detection of nickel in presence of over 1,000 times the amount of iron, preferably in ammoniacal citric acid solution. Thus 1 part of nickel and 1,200 parts of iron in 1,000,000 parts of water gave an immediate coloration and a much more voluminous precipitate than the dimethylglyoxime reagent, the colour of which only appeared after standing three hours.

The reaction with α-benzildioxime does not appear to be interfered with by the presence of large amounts of silver, magnesium, chromium, or manganese. The presence of large amounts of zinc has been found to prevent the formation of the nickel coloration with dimethylglyoxime, whereas the α-benzildioxime test is unaffected, a copious precipitate being obtained on standing a short time.

QUANTITATIVE APPLICATION OF THE REAGENT.

α-Benzildioxime forms a convenient reagent for the estimation of amounts less than 0·025 grm. of nickel. Owing to the rather low solubility of the reagent in alcohol, and the voluminous character of the precipitate, it is not advisable to have a larger amount of nickel present. A slight excess of a warm solution of the reagent in alcohol, to which ammonia has been added, is added, with stirring, to the ammoniacal nickel solution, and the whole heated on the water-bath for a few moments to coagulate the precipitate. Quantitative precipitation is complete after one minute, whereas in using the dimethylglyoxime reagent precipitation has often been found to have been incomplete after standing for an hour in a warm place. The liquid is filtered at the pump through a Gooch crucible or counterpoised filter-papers, washed with warm 50 per cent. alcohol, then with hot water, and finally dried at 110° C. The precipitate contains 10·93 per cent. nickel, and is hence very suitable for the determination of very small quantities of nickel. On washing with hot water the precipitate has no tendency to pass through the filter-paper, as is the case with the dimethylglyoxime precipitate (*c.f.* F. Ibbotson, *Chem. News*, 1911, **104**, 224). The compound is much less soluble in warm alcohol-water mixtures than that from dimethylglyoxime, whose solubility in alcohol-water mixtures was mentioned by Brunck (*loc. cit.*). Precipitation may also be effected with an acetone solution of the reagent, in which it is much more soluble.

The following are typical results obtained by the α-benzildioxime method, the standard solutions being made from recrystallised nickel ammonium sulphate

(Kahlbaum), which had been analysed by gravimetric methods, and found to be pure:

	Nickel Present. Grm.		Nickel Dioxime Precipitate. Grm.		Nickel Found. Grm.
1.	0·02748	...	0·2524	...	0·02758
2.	0·02269	...	0·2072	...	0·02266
3.	0·01645	...	0·1510	...	0·01650
4.	0·01645	...	0·1502	...	0·01642
5.	0·00480	...	0·0442	...	0·00481
6.	0·01535	...	0·1408	...	0·01539

Nos. 1 and 2 were weighed on asbestos in a Gooch crucible, Nos. 3 to 5 on counterpoised filter-papers, and No. 6 was precipitated by an acetone solution of the reagent.

As the use of counterpoised filter-papers is often adversely criticised, it was of interest to investigate whether weighing in this manner was to be considered accurate in this case. The filter-papers were 7 cm. hydrochloric acid washed Schleicher-Schüll papers, and these were subjected to identical treatment during the experiment with regard to washing, etc. In several of the above cases the precipitate was detached from the filter-paper into a tared weighing-bottle, a process which is much more easily accomplished in the case of the α-benzildioxime precipitate than that from dimethylglyoxime, only 0·2 to 0·4 mgrm. of the precipitate remaining on the paper. On reheating the filter-paper and weighing-bottle to 110° C., the total weight of the precipitate was not changed appreciably in any case, even after the temperature had been raised to 120° C. Exposure of the precipitate for half an hour in the balance-case showed that the precipitate does not absorb moisture to any appreciable extent. The method of weighing on counterpoised filter-papers is therefore available in this case.

The method is affected by the presence of nitrates, which must therefore be removed by evaporation with sulphuric acid before the addition of the reagent.

Ignition of the precipitate failed to yield satisfactory results, although care was taken to avoid loss of nickel by volatilisation of the compound as far as was possible. Possibly this is explained by the retention of small amounts of carbon, even though ammonium nitrate was added before the final ignition. P. Bogolubow (*Stahl u. Eisen*, 1910, **30**, p. 458) states that Wdowiszewski's method of igniting the dimethylglyoxime precipitate leaves carbon in the nickel oxide.

In Presence of Cobalt.—The method is available in presence of cobalt, precipitation being effected in ammoniacal solution. Owing to the formation of a soluble cobalt salt of the dioxime, excess of the reagent must be added, the precipitate being washed until the filtrate is colourless. The following results were obtained:

	Nickel Present. Grm.		Cobalt Present. Grm.		Nickel Found. Grm.
1.	0·00658	...	0·02	...	0·00661
2.	0·00480	...	0·04	...	0·00483
3.	0·01645	...	0·04	...	0·01648
4.	0·01134	...	0·02	...	0·01140

In No. 3 an acetone solution of the reagent was used.

In Presence of Iron.—Nickel can be estimated in presence of ferric salts in ammoniacal solution by keeping the iron in solution by Rochelle salt or, preferably, sodium citrate. A slight excess of the alcoholic solution of the reagent is added. The following results were obtained:

	Nickel Present. Grm.		Iron Present. Grm.		Nickel Found. Grm.
1.	0·01135	...	0·14	...	0·01130
2.	0·00567	...	0·03	...	0·00565
3.	0·01099	...	0·07	...	0·01102

The method is therefore available for the estimation of nickel in nickel steels.

In Presence of Manganese.—Precipitation is effected by a fairly large excess of the alcoholic reagent in slightly acetic acid solution. Satisfactory results were obtained.

In Presence of Zinc or Magnesium.—The zinc or magnesium is kept in solution by addition of a large amount of ammonium chloride, precipitation being effected in ammoniacal solution. The results showed that neither zinc nor magnesium interfere with the precipitation, and nickel may be estimated by α-benzildioxime in presence of these metals.

In Presence of Chromium.—Precipitation is effected in dilute ammoniacal solution in presence of tartaric acid, when the nickel present is precipitated quantitatively.

The further application of the method to the analysis of nickel alloys is at present being examined. Whereas small amounts of copper do not interfere with the method, larger amounts must be precipitated before addition of the reagent.

In conclusion, the author desires to express his indebtedness to Professor Knecht for the facilities afforded for the carrying out of the work.

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DISCUSSION.

Mr. E. R. **BOLTON** said that nowadays the question of the presence in fats of traces of nickel, palladium, and other metals, used as catalysts, was of considerable importance in dealing with hydrogenised or hardened fats. He had used the diacetyldioxime method quite successfully, but had found that, when the quantity of metal present was very small, the colour was fugitive, returning, however, subsequently. With diacetyldioxime, nickel could be separated from palladium, the palladium being filtered off from an acid solution, which was then made alkaline with ammonia, precipitating the nickel. He should be glad if the author would say where α-benzildioxime was to be obtained, as it was not included in the ordinary catalogues.

The **PRESIDENT** said that he used the dimethylglyoxime method frequently, especially for the determination of nickel in copper, and had found it very satisfactory. In that case the nickel was precipitated from a solution slightly acid with acetic acid, whereas, in the case of α-benzildioxime, he understood that the solution was ammoniacal. From what Mr. Atack had said, it appeared that the α-benzildioxime method was the more delicate, and gave a sharper separation between nickel and cobalt.

Mr. Atack said that the subject of hardened oils was now under investigation, but embraced some points which were rather troublesome. In a paper by Bömer (*Zeitsch. Untersuch. Nahr. Genussm.*, 1912, **24**, 104) it was said to have been found by Prall that certain fats gave the nickel reaction with dimethylglyoxime before any nickel had been added, but no details were given, and he (Mr. Atack) had not been able to confirm that, nor had he met with any case in which a hardened fat did not give the reaction with α-benzildioxime. He had not been aware of the process for the separation of palladium from nickel by dimethylglyoxime, though he understood that a process had been published by Wunder and Thüringer (*Zeitsch. anal. Chem.*, 1913, **52**, 101) for the separation of palladium from copper and iron, using this reagent. In the case of α-benzildioxime the palladium compound appeared to decompose rapidly, especially in the presence of light, but that point also was under investigation. α-Benzildioxime would probably be purchasable in the near future at cheaper rates than dimethylglyoxime, but even now it was quite easily prepared by boiling 10 grms. benzil (not necessarily pure) with 8 to 10 grms. of hydroxylamine hydrochloride in methyl alcohol solution. After boiling for three hours the precipitate is filtered off, dried, washed with hot water, and then a small amount of 50 per cent. alcohol, and dried, when it consists of pure α-benzildioxime (m.-pt. 237° C.). A further yield can be obtained by boiling the filtrate with hydroxylamine hydrochloride. He had tried precipitation from an acetic acid solution in the case of manganese, but care was necessary, or the α-benzildioxime would change into the β variety, which did not give the nickel reaction. Probably in the case of copper an acetic acid solution would have to be used, unless the copper were first separated by means of sulphuretted hydrogen.

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Detection of Aloes in Extracts of Drugs containing Oxymethylanthraquinones. G. Mossler. (*Pharm. Post.*, 1913, **46**, 313-315, 325-327; through *Chem. Zentralbl.*, 1913, I., 1842-1843.)—The alcoholic extract is evaporated to expel the alcohol, the residue taken up with water, and the liquid filtered. The filtrate (100 c.c.) is heated for thirty minutes on the water-bath with 5 c.c. of 10 per cent. sulphuric acid, the sulphuric acid precipitated with the exact amount of barium hydroxide solution, and the filtrate from the barium precipitate concentrated to 100 c.c. and clarified with lead acetate solution, care being taken to avoid a large excess. Ten c.c. of the solution are next filtered, and after removal of lead by means of sodium sulphate solution or dilute sulphuric acid, the filtrate is divided into two parts. One of these is shaken with 4 to 5 c.c. of benzene, and the benzene extract shaken with dilute ammonia solution. If the foreign oxymethylanthraquinones have been completely precipitated, the aqueous layer should not show more than a light rose colour, whilst a yellow coloration of the benzene layer indicates aloes. If, however, the aqueous layer is distinctly red, the bulk of the original solution must again be treated with lead acetate. When the precipitation has thus been shown to be complete, the other portion of the filtrate is treated with an excess of bromine water, which, in the presence of aloin, produces an immediate flocculent precipitate. The main solution is now freed from lead, and the filtrate tested by the reactions of Schouteten and Hirschsohn. In the former 10 c.c. of the filtrate are gently heated and shaken with 2 to 3 grms. of borax and allowed to stand for fifteen minutes, when, in the presence of aloes, a green fluorescence appears. In Hirschsohn's test 10 c.c. of the solution are heated with 1 drop of copper sulphate solution and 1 drop of hydrogen peroxide. In the presence of aloes a red coloration which becomes intensified on standing is obtained, whilst samples free from aloes become orange-red and do not alter on standing. In this way it is possible to detect 0·2 grm. of aloes extract in 5 grms. of a mixture of extracts of rhubarb, frangula, and cascara sagrada. For the identification of the different oxymethylanthraquinone drugs the alcoholic extract is brought to a strength of 50 per cent. of alcohol and filtered. The filtrate is boiled for thirty minutes beneath a reflux condenser with about 5 per cent. of sulphuric acid, and then cooled and filtered, the alcohol evaporated from the filtrate (with frequent additions of water), and the precipitated oxymethylanthraquinones separated from the liquid, any aloin remaining in solution. The precipitate is washed until free from acid, dried and boiled with benzene, and the benzene extract shaken with 10 per cent. sodium carbonate solution, and then with dilute sodium hydroxide solution. The two alkaline extracts are separately acidified with hydrochloric acid, each shaken with 20 c.c. of benzene, and 4 to 5 c.c. of the benzene extracts evaporated. The residues are heated with 3 to 4 drops of acetic acid, and the solutions transferred to glass slips and examined, after thirty minutes, in polarised light. The crystals thus obtained from rhubarb, cascara sagrada, senna, etc., show pronounced differences, as is also the case with the crystals obtained by sublimation.

C. A. M.

New Reaction for the Detection of Aniline Dyestuffs [Magenta] in Foods, and especially in Wines. P. Malvezin. (*Ann. Chim. anal. appl.*, 1913, **18**, 193.)—Methanal hydrosulphite, $CH_2OH.SO_3H$, obtained by passing sulphur dioxide into 40 per cent. formaldehyde solution, is coloured violet by a very dilute solution of magenta, the reaction being rendered more sensitive by warming. The wine to be tested is decolourised with animal charcoal, and 2 to 3 c.c. of the filtrate are shaken in a test-tube with an equal volume of the reagent, heat being applied if only a faint violet coloration is obtained. Under the same conditions wine free from aniline dyestuffs gives a faint rose coloration. C. A. M.

Estimation of Water in Cheese. Utz. (*Zeitsch. angew. Chem.*, 1913, **26**, 271-273.)—Results of numerous estimations of water in cheese by distilling the sample with petroleum and measuring the volume of water in the distillate, show that this method is trustworthy. It is recommended that the petroleum used should be that portion of ordinary paraffin oil which boils below 150° C. (*cf.* ANALYST, 1913, 146).

W. P. S.

Sudan Dura. (*Bull. Imp. Inst.*, 1913, **11**, 33-46.)—Dura, or "dari," is already imported into the United Kingdom, where it is chiefly used as poultry food, and generally fetches prices similar to those paid for maize. A sample of the grain previously freed from dirt had the following composition: Moisture, 8·45; crude proteins, 13·06 (true proteins, 12·98; other nitrogenous substances, 0·08); fat, 3·30; total carbohydrates, 72·4; starch, 62·66; fibre, 1·03; ash, 1·71 per cent.; food units, 113·3; nutrient ratio, 1 : 6·1. No alkaloids or cyanogenetic glucosides were found.

H. F. E. H.

Modified Babcock Test for Fat in Sweetened Ice Cream. J. O. Halverson. (*J. Ind. and Eng. Chem.*, 1913, **5**, 403-409.)—The Babcock test is not applicable to sweetened dairy products on account of the charring action on the sugar. It is noticeable that an appreciable interval of time elapses on adding sulphuric acid slowly before action on the sugar commences, and the modification suggested consists in draining off this acid sugar solution by using a special bottle fitted with a small glass stopcock (1 mm. bore) on one side of a 6·5 inch 25 per cent. cream test-bottle. To a volume of cream (17·6 c.c.) is added 7 to 10 c.c. of ordinary sulphuric acid in 3 c.c. portions. When the colour reaches an amber brown, 2 to 3 c.c. of cold water are added, with shaking to check the action. The whole is then centrifuged for five minutes, and then hot water is added nearly up to the neck, followed by a further two minutes centrifuging. The acid sugar solution is now drained off, and acid again added (about 8 or 10 c.c.) till a coffee-black colour is reached. Hot water is then added up to the neck, and the whole again centrifuged two minutes.

No further draining is necessary at this stage, and the fat is run into the neck with hot water, centrifuged one minute, and the reading taken. Very careful sampling of the ice cream is required in order to obtain consistent results. A number of results are recorded in comparison with the Rose-Gottlieb method, the modified Babcock test showing on the average results 0·6 per cent. low.

H. F. E. H.

"Cheyi" Seed. (*Bull. Imp. Inst.*, 1913, **11**, 62-65.)—The seed is derived from *Polygala butyracea*, a herbaceous plant common in tropical West Africa. The yield of fat is 38 per cent., having the following constants: M.-pt., 36° C.; sp. gr., $\frac{100^\circ\text{ C.}}{15{\cdot}5^\circ\text{ C.}} = 0{\cdot}866$. Acid value, 1·24; saponification value, 251·0. Iodine value, per cent., 52·5; Titer test, 37·85° C. Hehner value, per cent., 85·6; Reichert-Meissl value, 45·6. The stems of the plant yield a fibre considered by experts to be equal in value to fine Bombay hemp, and resembles Sunn hemp of similar appearance. H. F. E. H.

Oil Seeds from the Anglo-Egyptian Sudan. (*Bull. Imp. Inst.*, 1913, **11**, 56-62.)—Senat seed (otherwise "hameid," "fugus," "ajurr," and "tibish") is derived from various cultivated varieties of *Cucumis chate*, which is probably the wild type of the cultivated melon. All the samples yield a pale yellow liquid oil, free from smell or unpleasant taste. Six samples were examined, with the following results, to which are added for comparison the figures for oils from other cucurbitaceous seeds:

							Melon Seed (*Cucumis melo*).	Water-melon Seed (*Citrullus vulgaris*).
Yield per cent. ...	36·5	31·0	29·5	31·7	30·2	38·4	43·8	40·8
Sp. gr. at $\frac{15·5°}{15·5°}$ C.	0·923	—	—	0·923	0·925	—	—	—
Acid value* ...	1·0	—	—	0·7	0·7	—	—	—
Saponification value*	192·0	190·5	189·3	189·2	187·0	—	193·3	189·7
Iodine value per cent.†	117·0	121·3	117·0	124·0	128·5	—	101·5	118·0
Titer test. ‡ ...	30·3° C.	—	—	—	—	—	36° C.	32° C.

* Mgrms. of potassium hydroxide per grm. of fat.
† Method not described.
‡ Solidifying-point of fatty acids.

In the case of No. 1 the Hehner value was 96·6, the percentage of unsaponifiable matter 0·7, and the Reichert-Meissl value nil. The senat husks have only a very low feeding value, and contain much fibre and mineral matter, but their high ash (17 per cent.) contains 43 per cent. of potash (K_2O), which should render them useful as a manure.

Salvadore persica seeds yield 44 per cent. of a hard, bright yellow fat, with a faint, slightly unpleasant odour, having the following constants: Sp. gr. at $\frac{99° \text{ C.}}{15° \text{ C.}} = 0·867$; acid value, 9·3; saponification value, 245·2; iodine value per cent., 5·9; Titer test, 30·4° C. (approx.); m.-pt., 38° C.

H. F. E. H.

Table of Calculated Dry Substance of Milk by the Fleischmann Formula. R. Pfister and W. Leuze. (*Milchwirtschaftl. Zentralbl.*, 1913, **42**, 97, 134, 169, 196; through *Chem. Zentralbl.*, 1913, I., 1728.)—The values are calculated from the formula—

$$T = 1·2\,f + 2·665\left(\frac{100\,s - 100}{s}\right)$$

in which T is the total solids, and f the fat-content, both as percentages, and s the density at 15° C. The values of T may be read off directly from the table (published in the original journal) for values of f between 0·10 and 6·00, rising by 0·05, and for s from 1·0190 to 1·0400, rising by 0·0001.

O. E. M.

Preservation of Milk Samples for Analysis. G. Denigès. (*Ann. Chim. anal. appl.*, 1913, **18**, 189-192.)—A solution of 50 grms. of phenol in 10 c.c. of 95 per cent. alcohol has proved more satisfactory than any of the other antiseptic agents used for the preservation of milk samples. The addition of 1 c.c. of the solution to 100 c.c. of milk keeps the sample indefinitely, and does not affect the estimation of the acidity, lactose, fat, casein, ash, or extract. A sample of milk thus preserved gave exactly the same analytical results after ten years as at first. C. A. M.

Alteration of Milk treated with Potassium Dichromate. G. Hinard. (*Ann. Falsific.*, 1913, **6**, 233-237.)—Successive analyses, at intervals of a few weeks during a period of six months of a sample of milk which had been treated with potassium dichromate (0·98 grm. per litre) showed that the composition of the milk changed gradually, and that dichromate cannot be relied upon as a preservative of milk samples. In six months the total solids decreased to the extent of 10 grms. per litre, the solids-not-fat decreasing to about the same extent whilst the quantity of fat remained constant. The lactose varied considerably in quantity during the period, but at the end had diminished by 14 grms. per litre; the quantity of casein also decreased, whilst the acidity, expressed as lactic acid, increased from 2·3 to 6·6 grms. per litre. W. P. S.

Peroxydase Reaction of Milk. (Influence of Preservatives, etc.) T. Jona. (*Arch. Pharmacol. sperim.*, 1913, **15**, 122-130; through *Chem. Zentralbl.*, 1913, I., 1790-1792.)—Whilst the presence in milk of the usual preservative substances does not interfere with the peroxydase reaction, in certain cases the presence of the preservatives lowers the temperature at which the milk ceases to give the reaction when heated; for instance, milk containing salicylic acid or mercuric chlorides no longer gives the reaction when heated to about 69° C. As regards the estimation of the age of milk by means of the peroxydase reaction, the author finds that, although there is some relation between the age of milk (and its acidity) and its power of giving the reaction, definite conclusions cannot be arrived at from the indications yielded by the test. The addition of acetic or citric acid prevents the reaction with *p*-phenylenediamine, but the reaction with guaiacum is not affected; mineral acids and oxalic acid inhibit the reaction with both reagents, whilst the natural acidity of milk interferes to a less extent. With respect to the reaction mentioned by Gaucher (ANALYST, 1908, **33**, 237), it was found that a mixture of boiled milk with 40 per cent. of raw milk behaved like raw milk, as it no longer decolourises hæmatin. Raw milk, containing 0·2 per cent. of formalin at once decolourises hæmatin; milk containing traces of hydrogen sulphide behaves in the same way, but boiled milk to which hydrogen peroxide has been added does not decolourise the reagent. The presence of potassium dichromate or mercuric chloride interferes with the reaction.

W. P. S.

Estimation of Phenols in Creolins. A. J. J. Vandevelde. (*Bull. Soc. Chim. Belg.*, 1913, **27**, 128-129.)—Creolins are mixtures of hydrocarbons with alkali salts of phenolic bodies. The disinfectant value of the mixture can only be determined satisfactorily by bacteriological methods, but a determination of the total phenols is generally required. For this purpose, having found that certain of the hydrocarbons of creolin are not completely volatile with steam, the author prefers to acidify the mixture and to distil the phenols. Two hundred and fifty grms. of the creolin are treated with 500 c.c. of water and sufficient sulphuric acid to impart a distinctly acid reaction; the whole is placed in a separating funnel with 100 c.c. of benzene, and allowed to remain at rest for twenty-four hours. By the aid of the benzene the separation of the aqueous layer is easily effected, and the oily layer is then submitted to a simple distillation, collecting all the products distilling below 230° to 240° C. The distillate then contains the phenol, cresols, a portion of the more volatile hydrocarbons, and the added benzene. It is shaken three times in succession with quantities of 50 c.c. each of a 10 per cent. solution of sodium hydroxide. The aqueous portion, containing all the phenolic bodies, is separated and acidified in a graduated tube. The quantity of phenols may be ascertained approximately by dividing the volume of the layer by the mean density 1·055, or it may be separated and weighed. The results of duplicate estimations are in satisfactory concordance.

J. F. B.

Estimation of Saccharin in Foods. J. Karas. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1913, **25**, 559-560.)—In order to prevent contamination of the saccharin by other substances when the former is extracted from a food by means of ether, the following procedure is recommended: The essential feature of the process consists in precipitating interfering substances by the addition of tannin and lead acetate before the saccharin is extracted, the author having proved by experiment that saccharin is not precipitated by these reagents. The fluid under examination, or an aqueous extract of a food, is evaporated in order to remove any alcohol which may be present, and the residual liquid is treated with tannin solution (15 c.c. of a 10 per cent. solution per 100 c.c. of liquid) and 8 c.c. of basic lead acetate solution, and filtered. The filtrate is then acidified with phosphoric acid, the lead phosphate is separated by filtration, and the saccharin is extracted from the filtrate by means of a mixture consisting of equal volumes of ether and light petroleum. The residue obtained on evaporating the solvent consists of pure saccharin. When the substance under examination contains much fat, this may be removed by rendering the liquid alkaline, and extracting with ether before treatment with tannin and lead acetate.

W. P. S.

Melnik (Bohemian) Wine. F. Černý. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1913, **25**, 486-489.)—About one-half the quantity of wine produced in Bohemia is obtained from the province of Melnik. The vines are chiefly of the blue Burgundy variety, and were originally obtained from France. Analyses of thirty wines are recorded. The alcohol varied from 6·73 to 9·92; total solids from 1·71 to 3·82; glycerol from 0·65 to 1·24; acidity (tartaric acid) from 0·48 to 0·84; and ash from 0·14 to 0·39—all these results being expressed in grms. per 100 c.c. The wines were all free from sugar except in one case, "Château Melnik," where the quantity of sugar was 2·8 grms. per 100 c.c.

W. P. S.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Chlorine Content of Blood and its Distribution between Serum and Blood-Corpuscles. J. Snapper. (*Biochem. Zeitsch.*, 1913, **51**, 53-62.)—The author finds that if serum is treated with 2 to 3 parts alcohol, the alcoholic filtrate contains the total chlorine, whilst the precipitate is free from chlorine. The chlorine content of the corpuscles is from 40 to 45 per cent. of that of the serum. Assuming that the chlorine of the corpuscles is dissolved in the intraglobular liquid, this liquid is calculated to have the same chlorine concentration as the serum. E. W.

Residual Reduction of Blood. P. Mayer. (*Biochem. Zeitsch.*, 1913, **50**, 362-369.)—It is usual to estimate glucose in blood by measuring copper reduction and rotation, and correcting for other reducing and active substances by carrying out the same measurements after removal of glucose by fermentation. In order to determine whether this residual reduction is not partly due to substances formed during fermentation, 0·1 per cent. solutions of glucose were fermented with a number of pure culture yeasts. The results varied with the yeast used. In numerous instances the fermented liquid was active, mostly dextrorotatory, but sometimes also lævorotary, and many reduced Fehling's solution. The behaviour towards other reagents leaves no doubt that optical activity and reducing power are due to degradation products of yeast proteins, and not to unfermented glucose; and, therefore, that glucose solutions of concentrations corresponding to those occurring in blood may, after fermentation, exhibit rotatory and reducing power, which may lead to a false inference as to the nature of the substances to which the residual reduction is attributed. E. W.

Indicator Method for the Estimation of the Permeability of Cells by Alkalis. E. N. Harvey. (*Amer. J. Physiol.*, 1913, **31**, 335-342; through *Chem. Zentralbl.*, 1913, **1**, 1793.)—Results of an investigation carried out by the author show that the indicator neutral-red is equally sensitive towards ammonia and sodium hydroxide, even in the presence of proteins and lecithin, and that the indicator method is useful for the detection of alkalis in the interior of cells. Tubes containing egg albumin tinted with neutral-red, and others containing a mixture of egg albumin with proteins, starch, lecithin, caseinogen, vitellin, globulin, etc., were immersed in $\frac{N}{1000}$ solutions of ammonia and sodium hydroxide respectively. The rate of diffusion of the alkali into the albumin, as shown by the change in colour of the indicator, was the same in every case. Similar experiments were made in which diluted egg albumin tinted with neutral-red was shaken with lecithin dissolved in various solvents; the action of ammonia and sodium hydroxide on the resulting droplets of lecithin was then observed. When the lecithin was dissolved in benzene, xylene, toluene, turpentine, carbon disulphide, or carbon tetrachloride, ammonia at once penetrated the droplets, whilst sodium hydroxide did not do so. On the other hand, both alkalis penetrated the droplets at the same rate when the lecithin had been dissolved in ether, ethyl acetate, ethyl butyrate, ethyl propionate, or ethyl valerate; in this case a layer of albumin did not form round the droplets.

W. P. S.

Neutral Red Paper as Indicator for Alkalimetry of Serum. J. Snapper. (*Biochem. Zeitsch.*, 1913, **51**, 88-91.)—A concentrated solution of neutral red in 50 per cent. alcohol is diluted with 10 parts 50 per cent. alcohol. Filter-paper is soaked in this liquid and dried, and is prepared the same day that it is required. It is claimed to be superior to lacmoid in that it is not affected by proteins, and therefore gives a sharper end-point, and also that it can always be freshly prepared before use.

E. W.

Simple Method for Determining the Fermentative Activity of Milk and Various Feeding-Stuffs, and the Catalase Enzyme of Milk. A. Rosam. (*Milchwirtschaftl. Zentralbl.*, 1912, **42**, 193; through *Chem. Zentralbl.*, 1913, I., 1723.)—A modification of the Walter test is used. A weighed quantity, 1 to 5 grms., of the feed is warmed at 35° to 40° C. with water which completely fills a 50 c.c. tube shaped like an elongated conical flask, and connected by a rubber stopper to a rising tube. The level of the water is observed (1) twenty minutes, and (2) several hours after the tube is placed in the thermostat. As a standard, the fermentative activity which produces a rise of 30 cm. in three hours is taken as 100 per cent. ($\frac{30}{3} = 100$ per cent.). To determine the fermentative activity of feeding-stuffs on milk, sterilised milk is substituted for the water. The following figures were obtained: Sugar beet, 43 to 60 per cent.; fresh sliced roots, 30 to 50; sour sliced roots, 10 to 18; dry sliced roots, 25 to 40; fresh cabbage, dirty, 40 to 60; clover, 50 to 70; hay, 30 to 45; fresh brewers' grains, 0·5 to 5; used straw litter, 65; dung, 50 to 70; milk, 4 to 20. The maximum quantity of gas is formed at 30° to 40° C. To apply the method to the estimation of catalase in milk, 150 c.c. of milk are treated with 50 c.c. of 1 per cent. hydrogen peroxide, and warmed in the apparatus at 25° C.

O. E. M.

Composition of Milk from Cows suffering from Foot and Mouth Disease. O. Mesger, H. Jesser, and K. Hepp. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1913, **25**, 513-551.)—Systematic analyses of milk from twenty-six cows suffering from foot and mouth disease are recorded, the results showing that the influence of the disease on the composition of the milk varied more with the individual cow than with the breed or lactation period. At the commencement of the disease the yield of milk diminished, but the composition, as regards the fat and proteins, showed an improvement, the milk containing a larger proportion of these two constituents; an increase was sometimes noticed in the amount of ash, chlorine, and phosphoric acid contained in the milk.

W. P. S.

Estimation of Lactic Acid in Urine. H. Ishihara. (*Biochem Zeitsch.* 1913, **50**, 468-483.)—Ryffell's method (*Proc. Physiol. Soc.* 1909, **5**) modified by estimating the acetaldehyde iodimetrically according to Ripper (ANALYST, 1901, **26**, 131) estimates about $\frac{4}{5}$ of lactic acid present and is valuable as a rapid approximation, but cannot be used when sugar is present. For accurate work, lactic acid must be extracted with ether, but owing to its unfavourable distribution coefficient this cannot be done in a separating funnel. In order to avoid emulsification, the urine is first precipitated with phosphotungstic acid, then with baryta, the excess of baryta removed by carbon dioxide, concentrated and acidified with phosphoric acid. The resulting syrup is extracted for twenty-four hours in a Lindt extraction apparatus, and the lactic acid estimated according to the method of Fürth and Charnasy (ANALYST, 1910, **35**, 445), modified and simplified in some details. The method tested on urine with added lactic acid gave yields of 89 to 99, and a mean of 92 per cent. of the total acid. Normal human urine was found to contain 0·08 grm. lactic acid per litre. E. W.

Titration of Uric Acid in Urine after Silver Precipitation. E. Kretschmer. (*Biochem. Zeitsch.*, 1913, **50**, 223-233.)—Two hundred c.c. of urine are precipitated with magnesia mixture, made up to 300 c.c., filtered, and 200 c.c. of the filtrate precipitated with ammoniacal silver, as in Salkowski's process. The silver precipitate is washed until the filtrate is free from silver and chloride, transferred to a beaker, and decomposed with 15 to 20 c.c. concentrated sulphuric acid. The mixture is heated to boiling, immediately filtered from silver sulphate, and repeatedly washed with boiling water. The solution (about 250 c.c.) is cooled to 60 to 64° C., and immediately titrated with $\frac{N}{20}$ permanganate. The filtration from the silver sulphate is essential, but the slight opacity that is sometimes observed in the filtrate does not interfere with the titration. The somewhat high results obtained as compared with those given by Salkowski's method are probably due to the purin bases carried down in silver magnesium precipitate which are also oxidised; they cannot be attributed to urea, since even a 0·1 per cent. urea solution is not oxidised by $\frac{N}{20}$ permanganate. E. W.

ORGANIC ANALYSIS.

Studies of the Conditions of the Precipitation of Albumin by Picric Acid. H. Labbé and R. Maguiss. (*Comptes rend.*, 1913, **156**, 1415-1417.)—The precipitates formed with albumin by various reagents are generally of the type of adsorption compounds, or at any rate are readily dissociated on washing, so that they do not lend themselves to gravimetric estimations. The relations between the proportions of the reacting substances also are not constant, but are only represented by more or less complicated curves. Denigès and, later, Vallery have worked out a method for the estimation of albumin by precipitation with potassium mercury iodide, employing a curve for the expression of the results. The authors have now investigated a simple volumetric method for the estimation of egg albumin by precipitation by the citro-picric acid reagent prepared according to Esbach. If a fairly large excess of picric acid be used, the precipitation of egg albumin is complete and the clear filtrate is free from albumin. Starting with a known quantity of picric acid, the quantity of acid remaining in the filtrate gives a measure of that fixed by the precipitate. With increasing quantities of albumin and a constant quantity of picric acid, a series of numbers are obtained which may be plotted on a curve. Fifty c.c. of Esbach's citro-picric acid reagent are diluted to 100 c.c., and the acidity is determined by titration with $\frac{N}{10}$ sodium hydroxide in presence of phenolphthaleïn. Increasing volumes of a 10 per cent. solution of egg albumin are successively mixed with 50 c.c. of the picric reagent and diluted to 100 c.c. The excess of acid is then titrated on 25 c.c. of the filtrate. If the quantities of acid fixed expressed in c.c. of $\frac{N}{10}$ alkali be represented by y and the quantities of albumin taken by x, a curve may be constructed, which for values of x between 0·8 and 2·60 grms. is practically a branch of a hyperbola, adequately represented by the formula—$x = \frac{76{\cdot}08}{18{\cdot}24 - y} - 4$. Such a curve is characteristic of adsorption compounds, and it has been found that the albumin-picric acid precipitate is dissociated by washing at a decreasing rate as the amount of picric acid remaining decreases, so that the last traces are held very firmly by the albumin. With the use of the curve above mentioned, the estimation of egg albumin may be effected by two simple acidimetric titrations. J. F. B.

Estimation of Methyl and Ethyl Alcohols in the Presence of Each Other. J. Meyerfeld. (*Chem. Zeit.*, 1913, **37**, 649-651.)—The method is based upon the same principle as that devised by Hehner (Analyst, 1887, **12**, 25). The mixture of the two alcohols is oxidised by heating it with a measured quantity of chromic acid solution in the presence of sulphuric acid, and the excess of the oxidising agent titrated with standard thiosulphate solution. C. A. M.

Sampling Coal Deliveries. G. S. Pope. (*U.S. Bureau of Mines, Bulletin* 63, pp. 68.)—Anthracite larger than pea size is now bought by the United States Government on a guaranteed maximum percentage of ash on the dry coal. If this is exceeded, the Government has the option of refusing delivery or deducting 10 per cent. from the bill. Anthracite peas and smaller sizes are bought on a guaranteed maximum percentage of ash and minimum heat value, both calculated on the dry coal. Coal which is not up to specification may be refused, or 25 per cent. deducted from the price at the purchaser's option. Bituminous coal is bought on a guaranteed maximum percentage of ash, volatile matter and sulphur, and minimum heat value, all calculated on the dry coal, and on a guaranteed maximum percentage of moisture in the coal as received. The determination of moisture is only made when the sampler suspects an undue percentage, and in such cases he is required to collect a special moisture sample with the utmost speed and without the refinements adopted to make the ordinary sample uniform, as such refinements would, by loss of moisture in the process, introduce errors, beside which the slightly more representative character of the sample would be negligible. These moisture samples are of about 30 to 50 pounds, and are rapidly crushed to ½ inch and quartered down to 3 pounds. The ordinary sample is collected with the utmost care to make it representative and without regard to moisture losses in the process, as the price to be paid depends directly on the heat value of the dry coal, and is subject to increase or decrease according to an agreed scale for every 1 per cent. that the ash is less or more than that named in the contract. It is laid down in the contracts that the gross sample shall be not less than 600 pounds in the case of slack and small anthracite, nor less than 1,000 pounds with larger coal, and it is shown in this bulletin that there is rarely any advantage in collecting a gross sample larger than 1,500 pounds. Detailed directions are given for mixing and quartering such quantities. When prepared by hand, the pieces of coal and impurities are crushed to the approximate size indicated in the table below before each reduction :

Weight of Sample to be Divided.	Size to which Coal and Impurities should be Broken before each Division.
1,000 pounds or more	1 inch
500 pounds	$\frac{3}{4}$,,
250 ,, ...	$\frac{1}{2}$,,
125 ,, ...	$\frac{3}{8}$,,
60 ,, ...	$\frac{1}{4}$,,

The $\frac{1}{4}$-inch mesh sample is then reduced to about 3 pounds by quartering or, preferably, by the use of a mixing and reducing machine. G. C. J.

Note by Abstractor.—Formerly the United States Government purchased coal on an "as received" basis. The change to the "dry coal" basis, and the reasons given for it on pages 8 and 9 of this bulletin, will disappoint those who believe that coal, like other commodities, should be paid for at a rate proportioned to its value to the user, and will be held by those who oppose that principle to give away the whole case for purchase to a specification controllable by scientific methods and not by rule-of-thumb arbitration.

On pages 13 and 14 scathing comments are passed on specifications which provide substantial penalties for excessive volatile matter and sulphur. The Bureau of Mines only uses the percentage of sulphur and volatile matter to classify and identify coals, and this may serve their purpose. But a 2 per cent. excess of volatile matter in the case of anthracite would probably indicate substitution, and not improbably substitution of a coal which would gum up a gas engine in half an hour if run on gas ignition, or still more hopelessly, but after a longer run, if fitted with appliances for electric ignition. Again, though it may be admitted that sulphur has less to do with clinkering difficulties than was formerly supposed, and that its tendency to give trouble of this kind can be overcome by simple means, the writer might have reflected that coal is used for other purposes besides firing boilers. His responsibility is very great, because his department led the way in the past in the purchase of fuel to specification, and future utterances of the department on the subject will be given weight for that reason and without any necessary reference to their intrinsic value.

Studies on Cellulose. E. G. Parker. (*J. Phys. Chem.*, 1913, **17**, 219.)—The estimation of cellulose depends upon its resistance to attack by ordinary reagents, which remove the accompanying non-cellulose substances. In Lange's method this is effected by fusion with potassium hydroxide and water; small variations in the procedure produce considerable variations in the results, but the chief defect lies in the constantly varying concentration of alkali, due to the loss of water by distillation. The method was therefore varied to the following: 0·5 to 2·0 grms. of the sample, of known moisture-content, were heated to 130°-140° C. with 50 c.c. of potassium hydroxide solution in a 500-c.c. round flask, by means of a paraffin-bath. When it had been heated for the desired time, the cellulose was washed with water, and then, on a Gooch crucible, with dilute acid, dilute alkali, water, alcohol, and ether; it was then dried and weighed. With a 20 per cent. solution of the alkali, constancy was reached in fifteen hours; with a 10 per cent. solution, in four or five hours; with lower concentrations, down to the limit studied, the time is decreased as the dilution. The method was confined to surgical cotton-wool, which was found to contain 92 to 93 per cent. normal cellulose, 4-5 per cent. of cellulose, etc., soluble in potassium hydroxide, and 3·25 per cent. moisture. Cotton reprecipitated from Schweitzer's reagent gave irregular results; further study of the reaction is thus necessary. O. E. M.

Rapid Method of Testing Suction Gas Fuels for Liability to Clog Gas Engine Valves. W. McD. Mackey. (*J. Soc. Chem. Ind.*, 1913, **32**, 523-524.)—It is pointed out that the percentage of volatile combustible matter in anthracite is no certain indication of the behaviour of the fuel as a source of producer gas for power purposes. Though a low percentage of volatile matter usually connotes a satisfactory fuel, and a high percentage rightly engenders suspicion, it may happen that of two coals not differing greatly in their content of volatile matter, that with the larger amount may prove satisfactory in use, whilst the other gives rise to trouble. It is suggested that the quality rather than the quantity of the volatile matter determines the difference, and the following test for volatile substances of relatively high boiling-point is recommended: About 3 grms. of the powdered coal is heated in a platinum crucible for seven minutes, the bottom of the crucible being just above the tip of the inner flame of a Bunsen burner. The crucible is supported in a ring of asbestos cardboard, and covered by a 4-inch clock-glass containing 20 c.c. of water. If no tar is deposited on the clock-glass, the fuel may be considered satisfactory for use in a gas producer with the minimum provision for scrubbing the gas which characterises suction gas plants. G. C. J.

Detection of Gelatin in Textile Dressings. E. Schmidt. (*Färber-Zeit.*, 1913, **24**, 97-98; through *Chem. Zentralbl.*, 1913, I., 1896.)—Ammonium molybdate yields a flocculent white precipitate with gelatin solutions; the precipitate dissolves partially when the mixture is heated, but forms again on cooling. Another sensitive reagent for gelatin is prepared by rendering Nessler's reagent feebly acid with sulphuric acid and filtering off the red precipitate which is produced; the yellow filtrate yields a white turbidity when added to a gelatin solution, either hot or cold. Ammonium salts do not interfere with the reaction, but proteins other than gelatin must not be present. These two tests will detect as little as 0·01 mgrm. of gelatin in 5 c.c. of solution, a quantity which cannot be detected by the biuret or tannin tests. The substance under examination should be freed from fat and from albumin by means of nitric acid before the tests are applied, and alkaline solutions must be neutralised previously. W. P. S.

Detection of Hydrocyanic Acid. C. Pertusi and E. Gastaldi. (*Chem. Zeit.*, 1913, **37**, 609-610.)—The solution to be tested for hydrocyanic acid is added drop by drop to a reagent prepared by mixing together 1 drop of 3 per cent. copper acetate solution, 1 c.c. of 10 per cent. disodium phosphate solution, and 4 drops of a saturated solution of benzidine acetate. A turbidity followed by a bluish-violet coloration is obtained if the liquid contained as much as 0·000027 grm. of hydrocyanic acid per c.c., whilst the phosphate prevents the analogous colorations otherwise given by iodides, ferrocyanides, etc., with benzidine acetate and copper acetate. A sensitive modification of the test consists in boiling the solution with sodium hydroxide for five to ten minutes, filtering it and passing a current of carbon dioxide through the filtrate. The liberated hydrocyanic acid is carried onwards into the reagent, which for this purpose consists of a mixture of 1 drop of 3 per cent. copper acetate solution, 5 drops of benzidine acetate solution, and 0·5 c.c. of water. Under these conditions a distinct coloration is obtained in the presence of 0·000007 of hydrocyanic acid (as cyanide) in 10 c.c., whilst no reaction is obtained with chromates, permanganates, persulphates, periodates, ferric chloride, etc. In the presence of metals which form double cyanides soluble in solutions of alkalis, a preliminary separation of the cyanides as silver salts from the bases is necessary, the test being then applied to the precipitate. Schönbein's benzidine reaction with guaiacum tincture in the presence of a copper salt shows an analogous behaviour. If the tincture be freshly prepared and an addition of disodium phosphate made, the appearance of the colorations produced by bromides, iodide, and thiocyanates (but not cyanides) is prevented. (*Cf.* Lander, ANALYST, 1911, **36**, 266.)

C. A. M.

Evaporation Test for Mineral, Lubricating, and Transformer Oils. C. E. Waters. (*J. Ind. Eng. Chem.*, 1913, **5**, 394-398.)—It is shown by a series of parallel estimations that the results obtained in the evaporation test for lubricating oils are influenced to a great extent by the size of the vessel. For example, an oil heated for various periods in a vessel 2 × 3 cm. in size showed an average loss of 0·23 per cent., whereas when heated under the same conditions in a vessel 5 × 3 cm. in size, it gave an average loss of 0·63 per cent. Hence, in order to obtain comparable results, the same weight of oil must always be heated in vessels of the same size. Brass is more suitable than glass as the material for the vessel, and it is suggested that convenient dimensions are 5 cm. internal diameter, and height 3 cm. A vessel may be made from brass tubing with walls 0·75 mm. thick, and the bottom of sheet brass not exceeding 0·5 mm. in thickness should be soldered to this with silver solder. In making an estimation, the oil (5 grms.) should be weighed with the aid of a pipette and rod into this vessel without smearing the sides.

C. A. M.

Estimation of Pentoses in Presence of Other Sugars by Means of the Spectroscope. E. Pinoff and K. Gude. (*Chem. Zeit.*, 1913, **37**, 621.)—Tollens' phloroglucinol-hydrochloric acid reaction for the detection of pentoses may be modified so as to serve as the basis of a quantitative method. A permanent coloration may be obtained by heating the reagents in alcoholic solution, and the diluted solution shows one to three absorption bands, the intensity of the colour being proportional to the quantity of pentose taken. In a flask of 150 to

200 c.c. capacity, fitted with a reflux condenser, are placed 25 c.c. of the aqueous pentose solution, which should not contain more than 3 per cent. of pentose, 25 c.c. of hydrochloric acid of sp. gr. 1·19, 50 c.c. of 96 per cent. alcohol, and 0·6 grm. of phloroglucinol. The mixture is heated on the water-bath for half an hour, counting from the time the alcohol begins to boil. The flask is quickly cooled to avoid loss of alcohol, a portion of the solution is measured out and placed in a Hehner's colorimeter cylinder with the addition of so much alcohol that the spectroscope just shows faintly one or two of the characteristic absorption bands in the red and the yellow regions. The degree of dilution required to effect this result, *i.e.*, the number of volumes of alcohol added to the coloured solution, *plus* 1, multiplied by 0·0948, gives the percentage of pentose in the original solution. An original solution containing 0·0948 per cent. of pentose, 25 c.c. of which are treated according to the above method, when placed in a Hehner's cylinder in front of the spectroscope, will just show the bands with the limiting degree of intensity without any dilution with alcohol. A Hehner's cylinder is a graduated vessel of exactly 3 cm. internal diameter; any other cylinder of the same diameter may be used for the test. Other sugars do not interfere with the results, and since the method is a spectroscopic and not a colorimetric one, coloured solutions do not interfere. The method has been satisfactorily applied for the estimation of pentose in urine, and the experimental error does not exceed 0·1 per cent., even when the liquids contain over 2 per cent. of pentose. J. F. B.

Analysis of Paints and Especially Lithopones. P. Nicolardot. (*Ann. Chim. anal. appl.*, 1913, **18**, 184-186.)—The paint, etc., is treated with hot toluene to separate the oil from the mineral matter, the latter attacked with dilute hydrochloric acid beneath the surface of the toluene, and the liberated hydrogen sulphide conducted through two absorption vessels containing fuming nitric acid, followed by vessels containing sodium hydroxide and potassium permanganate solution respectively. Five grms. of the sample are weighed into a small tube, which is introduced into the reaction flask, the outlet of which is connected with the absorption vessels. An addition of 50 c.c. of toluene and 150 c.c. of hot water is then made, and after separation of the oil, 5 c.c. of hydrochloric acid are introduced little by little, and the contents of the flask heated nearly to boiling-point. When the reaction slackens, a slow current of air is passed through the apparatus, and the heating continued for about two hours until hydrogen sulphide is no longer evolved. The toluene layer is then filtered, the filter washed with hot toluene, the filtrate and washings dried over potassium carbonate and evaporated, and the residue of oil dried at 105° C. and weighed. The lower layer in the flask is also filtered, and the insoluble residues on the two filters are dried, ignited, and weighed (barium sulphate, silica, etc.). Zinc sulphate is estimated in the acid solution, and the sulphur from the oxidation of the hydrogen sulphide is estimated in the contents of the first three absorption vessels, the permanganate solution in the fourth vessel being used to show that the absorption in the other vessels was complete. Should the fourth vessel contain any sulphate the estimation should be repeated more slowly. C. A. M.

Preparation of Specifications for Petroleum Products. I. C. Allen. (*U.S. Bureau of Mines, Technical Paper* 36.)—In its efforts to increase efficiency in the utilisation of petroleum products, the Bureau has undertaken the preparation of specifications for certain petroleum products purchased by the Government. The task is a difficult one, and requires for its adequate performance much information which is not yet available. Recognising the importance of the work undertaken by the International Petroleum Commission, the Bureau has promoted co-operative research, in which its own staff, the Petroleum Commission, the American Chemical Society, the American Society for Testing Materials and other bodies, and private chemists and engineers are taking part. A list is given of thirty fundamental factors to be considered in petroleum testing, and of fourteen classes of products for which it is desirable to prepare specifications. G. C. J.

Effect of Temperature, Acid Concentration, and Time in the Bromination of Phenol for Quantitative Determinations. L. V. Redman, A. J. Weith, and F. P. Brock. (*J. Ind. and Eng. Chem.*, 1913, **5**, 389-393.)—The solutions employed are $\frac{N}{10}$ sodium thiosulphate, $\frac{N}{10}$ bromide-bromate (2·76 grs. potassium bromate, and 15 grms. bromide per litre), 20 per cent. potassium iodide, and 0·5 per cent. starch solution. The bromide-bromate solution is standardised iodimetrically against the thiosulphate solution. Into a 500 c.c. glass-stoppered bottle are placed 50 c.c. water, 5 c.c. hydrochloric acid (sp. gr., 1·2), and 15 c.c. of the phenol solution. This last, if necessary, should be diluted to $\frac{N}{10}$ strength. Sufficient $\frac{N}{10}$ bromide-bromate solution is now added while slowly shaking to give a slight permanent yellow coloration. The temperature at this point should be about 22° C. The stopper is replaced, and the whole shaken continuously for one minute, after which 0·5 c.c. potassium iodide solution is added, the bottle again shaken for a minute, and the titration finished with thiosulphate, the starch indicator not being added until the colour is almost discharged. Each c.c. of bromide-bromate solution is equivalent to 0·00156 grm. phenol. The results show that phenol may be rapidly determined as tribromophenol by bromination to within an error of 0·00005 grm. The quantities of reagents as generally recommended may be decreased with advantage. Two per cent. excess of free bromine is sufficient for the complete bromination of phenol in one minute in $\frac{N}{2}$ acid solution at 22° C. A large excess (over 50 per cent.) of potassium iodide above that necessary for complete reduction of the free bromine according to the equation is not required. It is shown that not more than 50 per cent. excess potassium bromide over that called for by the equation

$$KBrO_3 + 5KBr + 6HCl = 6KCl + 3B_2r + 3H_2O$$

need be used in making the standard bromide-bromate solution. The amounts recommended by the U.S. Pharmacopœia demand an excess of 340 per cent.

Complete liberation of the iodine by the free bromine may be effected in one minute if thorough diffusion be obtained by sufficient shaking. The acidity of the solution in which the tribromophenol is precipitated must not fall below 0·48 normal if the bromination is to be complete in one minute; an acidity of 0·5 to 1·0 normal is advised. Low temperatures retard the reaction; 20° to 30° C. is recommended (*cf.* ANALYST, 1912, **37**, 515).

H. F. E. H.

Estimation of Phenol in Presence of Organic Matter. E. M. Mumford.

(*Chem. News*, 1913, **107**, 253.)—Ordinary methods for estimating phenol are unsuitable when applied to solutions which have been subjected to bacterial action and contain large quantities of peptone and other proteins. The method adopted is based on the principle that phenol-sulphonic acid is readily nitrated, and can then be converted into ammonium picrate by the action of ammonia, and colorimetrically estimated. A suitable volume of the phenol-containing liquid is warmed with a few c.c. of concentrated sulphuric acid to 80° or 90° C., and into the warm liquid is then run a volume of 10 per cent. potassium nitrate sufficient to oxidise the organic matter present, and at the same time the phenol-sulphonic acid will be nitrated. The whole is then warmed, and if necessary boiled, to destroy the organic matter, until the liquid is straw-yellow or colourless. When cool and rendered alkaline with concentrated ammonia, the colour is matched against a standard solution of phenol-sulphonic acid which has been nitrated and made alkaline in a similar manner. Extreme accuracy is not claimed for the method, but 0·0001 grm. phenol can be thus estimated. The method is applicable to the estimation of α and β-naphthols.

H. F. E. H.

Report on the Present State of Rubber Analysis. F. W. Hinrichsen.

(*Sixth Congress Int. Assoc. Testing Materials, New York*, 1912; through *Chem. Engineer*, 1913, **18**, 165-166.) — Some international agreement on the methods of rubber analysis would be desirable. The analysis of crude rubber should preferably always be made on the mechanically washed, air-dry material. The moisture is determined by drying a portion of the sample *in vacuo* over sulphuric acid at a gentle heat. For estimating the resin, 5 grms. of the sample, finely-chopped, are extracted with acetone in a Soxhlet apparatus for ten hours; it is recommended that the body of the extraction apparatus be made of brown glass to avoid any action of the light on the material under treatment. The dimensions of the apparatus should be agreed upon. Direct methods for the estimation of caoutchouc are unreliable; this constituent is determined by difference after estimating the ash and proteins (N × 6·25). The analysis of vulcanised rubber mixtures is often very difficult, so that the united German cable manufacturers have adopted for standard insulations only mixtures which contain matters which can be determined by analysis—viz., in addition to caoutchouc and sulphur, only inorganic matters and hydrocarbons of the paraffin series (ceresin). When the mixture contains pitch, tar, asphalt, and the like, a satisfactory analysis is impossible. In many cases the following scheme is recommended: Five grms. of the sample are extracted with acetone, as in the case of crude rubber. This treatment will dissolve, in addition to the original rubber resins, any resins possibly added, free sulphur, oils, hydrocarbons of the paraffin series, and wax. The sulphur is determined in the evaporated acetone extract of a separate sample by oxidation with nitric acid and bromine. Solid hydrocarbons are approximately estimated by taking up the evaporated acetone extract with alcohol, and cooling this solution to − 5° C. When oils and foreign resins are present, the accurate estimation of the rubber resins is not possible; otherwise they may be calculated by difference. The saponification value has little or no significance on account of the variable nature of the resins. The material left after extraction with acetone is boiled for four hours under a reflux condenser with paraffin oil of high boiling-point, which dissolves the rubber and allows the filling matters to be filtered off or removed by centrifuging after the addition of benzene. Another portion of the material after extraction with acetone is boiled for four hours with $\frac{N}{2}$ alcoholic potassium hydroxide; this saponifies the usual substitutes; the acids of these are precipitated and separated by ether. Many of the substitutes used will have already yielded portions soluble in acetone. The total sulphur is estimated by oxidation with nitric acid and magnesium nitrate in a spherical flask, or else by electrolytic oxidation in nitric acid. From this is deducted the free sulphur (soluble in acetone), the sulphur combined with inorganic fillers and the sulphur in the substitute fatty acids; the difference represents the sulphur combined with caoutchouc. Lastly, the moisture and the protein are estimated, and the balance is expressed as caoutchouc. J. F. B.

Direct Determination of Rubber. L. G. Wesson. (*J. Ind. and Eng. Chem.*, 1913, **5**, 398.)—The procedure consists in allowing the acetone-extracted sample to dissolve or swell up in carbon tetrachloride, after which nitrous gases are passed in to form the nitrosite of rubber. The composition of the nitrosite is immaterial so long as it contains all the carbon of the rubber. After standing, the soluble nitrosite is dissolved in acetone, from which it is obtained by a method of precipitation or evaporation, in a form ready for combustion. The percentage of carbon so found is a measure of the rubber originally present. A special electric combustion-furnace is employed, and the material is burnt in a boat made of lead peroxide and red lead. The use of this boat, which absorbs the sulphur of the nitrosite as lead sulphate, should also give a means for the estimation of the sulphur of vulcanisation if, as seems probable, the sulphur combined with the rubber is carried quantitatively into its nitrosite. Some selected analyses give excellent results, but further work is required to test the reliability of the method and to eliminate the numerous sources of error encountered. H. F. E. H.

Use of the Köttstorfer Value for the Estimation of Resins and Substitutes in Caoutchouc Articles. M. Vallery. (*Monit. scient.*, 1913, **3**, 82-83; *Chem. Zentralbl.*, 1913, I., 1634-1635.)—More simply than by the usual method of extraction with alcoholic sodium hydroxide, the percentage of resins and substitutes may be estimated by determining the Köttstorfer value of the sample—viz., the quantity of potassium hydroxide in mgrms. absorbed by 1 grm. of the sample. The results, though not exact, are sufficiently good, and the method possesses the advantage that it is not influenced by the inorganic loading materials, so that a previous treatment with acetic acid is unnecessary. A weighed quantity of the rasped sample is boiled under a reflux condenser for three hours with such a quantity of alcoholic $\frac{N}{1}$ sodium hydroxide solution that the decrease in alkalinity is at least one-fifth of the original strength. After cooling, a slight excess of standard hydrochloric acid is added, and the excess is titrated back with $\frac{N}{10}$ sodium hydroxide in presence of phenolphthaleïn. From the quantity of sodium hydroxide consumed during the boiling the Köttstorfer value is calculated. It is necessary to correct the result by a blank experiment under the same conditions. Duplicate estimations should not differ by more than 3 units. The author has investigated the influence on the results of the state of subdivision of the sample. Estimations were made using cubes of various sizes of the same sample. The results showed that the results obtained when using cubes were very much lower than with the rasped material. Moreover, they increased at a slower rate than the total surface-area of the cubes. Thus the alkali does not act only on the surface of the particles, but penetrates to some extent. However, the equilibration of the concentration between the surface and the interior appears to take place very slowly, so that the action of the alkali on the cubes of cut caoutchouc is only very incomplete as compared with its action on the rasped material. Hence the use of rasped material is essential. J. F. B.

Maumené Figure of Oil of Turpentine. C. Grimaldi and L. Prussia. (*Chem. Zeit.*, 1913, **37**, 657.)—A reagent is prepared by running from a burette 1 part of sulphuric acid (sp. gr. 1·84) into 7·5 parts by volume of cold iso-amyl alcohol. Twenty c.c. of the oil are pipetted into a Dewar's vacuum-tube, the temperature taken with a stirring thermometer, 10 c.c. of the freshly prepared reagent introduced, and the rise of temperature noted. In the case of pure fresh oils of turpentine (French, Spanish, Portuguese, Austrian, Greek, and American) the values ranged from 77 to 85·7, and remained practically constant after distillation of the oils over lime. The fraction of the oils boiling between 155° and 156° C. (corresponding to the pinene) gave Maumené figures varying from 76·7 to 83·4, even after distillation over metallic sodium. On the other hand, oxidised oils gave much higher values (sometimes over 100), and the mixture turned brown, but after distillation over metallic sodium the Maumené figures of the original oils were obtained. Pine oils of various origins gave results varying within wide limits, but never exceeding 47·2; whilst *pinoline* and rosin essence (Austrian and American) gave a maximum value of 30·1, and petroleum derivatives and tar oils (benzene, zylene, toluene) showed practically no rise of temperature. In the case of pine oils and rosin essences the rise in temperature was increased by starting from a higher temperature—*e.g.*, 25° C.—but turpentine oil values were not materially affected by this factor. The method should detect relatively small quantities of petroleum spirit and pine oil in oil of turpentine. C. A. M.

INORGANIC ANALYSIS.

Preparation of Petermann's Ammonium Citrate Solution. F. Zöhren. (*Chem. Zeit.*, 1913, **37**, 611-612.)—Petermann's solution (ammonium citrate) may be prepared without loss of ammonia by adding the ammonia to the citric acid in a small filter-flask provided with a side tubulure. The cork of this flask has two openings, through one of which passes a stirring device worked by a small motor, whilst the ammonia solution is added through a dropping funnel, the end of which delivers below the surface of the liquid. The place where the stirrer passes through the cork is made secure by a piece of rubber-tubing drawn over the inner part of the stirring rod, and having its end dipping below the liquid. The flask is cooled in a bath of running water, and its side tubulure is connected with a small washing bottle containing a small weighed quantity of citric acid dissolved in a little water. This is subsequently added to the solution in the mixing flask, and its weight is taken into account. C. A. M.

Rapid Methods for Estimation of Antimony. H. Nissenson. (*Zeitsch. anorg. Chem.*, 1913, **81**, 46-48.)—No new methods are described, but alternative rapid methods for both sulphide and oxidised ores are given in sufficient detail, with references to the original papers in which they were first described. With the exception of the electrolytic method, no method more than ten years old is recommended. G. C. J.

Purification of the Precipitate obtained in Estimating Barium as Sulphate. F. A. Gooch and D. U. Hill. (*Zeitsch. anorg. Chem.*, 1913, **80**, 397-401.)—The paper relates to elimination of alkali salts by treatment with concentrated sulphuric acid and evaporation to dryness, which treatment results in the formation of a coarse crystalline precipitate, from which the alkali salts are easily washed out. The evaporation can be completed in half an hour if a Hempel burner is available, but occupies several hours, and requires much care, if an ordinary ring burner is used. The authors tried directing a blowpipe flame on the surface of the liquid contained in a crucible, but as erratic results were sometimes obtained, they recommend the following arrangement: A cone of fine platinum gauze, with a base somewhat greater in diameter than the crucible containing the liquid, is inverted in the crucible; the blowpipe flame is then directed against the apex of the cone, which should not touch the liquid. Five c.c. of sulphuric acid can be evaporated without loss in half an hour. The maximum error of a barium determination conducted in this manner is ±1·5 mgrms. on a weighing of 0·5 grm. The test precipitations were made in presence of 2 grms. of potassium chloride. Without purification of the precipitate, the average error was over +1 per cent. G. C. J.

Contribution to the Study of the Colour Reactions of Chlorates. M. E. Pozzi-Escot. (*Bull. Soc. Chim.*, 1913, **13**, 498.)—To 1 c.c. of the chlorate solution is added two drops of aniline sulphate solution (1 in 10), and 3 or 4 c.c. of concentrated sulphuric acid are poured down the side of the tube. At the common surface of the liquids a blue colour is formed if 0·00005 grm. of chlorate is present. If benzidene sulphate is used instead, an orange-yellow colour shows the presence of 0·000005 grm. of chlorate. O. E. M.

Influence of Chlorine on Determination of Nitrates by Phenol Disulphonic Acid Method. R. Stewart and J. E. Greaves. (*J. Amer. Chem. Soc.*, 1913, **35**, 579-582.)—The influence of chlorides on the determination of nitrates by the Grandval and Lajoux method is well known, but it is not generally recognised that the use of chloroform, to inhibit the action of bacteria in soil extracts, may result in the formation of ionic chlorine in amount sufficient to cause an error of 50 per cent. in a nitrate determination. This is especially the case when the lime method is employed, which method is recommended for use with soils rich in organic matter, since a clear solution is thus obtained; but in such cases chloroform should not be used. With 1 grm. of lime and as little as 2 drops of chloroform to 50 grms. soil, after standing overnight, small quantities of nitrate may be underestimated by more than 50 per cent., and even very large quantities by 20 per cent. Experiments are described which show that the above errors are due to the formation of chlorides, as a result of the decomposition of chloroform by lime. G. C. J.

Titration of Chlorine Bleaches. F. Ducelliez. (*Bull. Soc. Chim.* 1913, **13**, 494.)—The author has worked out a method depending upon the evolution of oxygen in presence of cobalt peroxide. For the valuation of chloride of lime, 2 grms. of the sample are heated with 0·5 grm. of cobalt chloride in a tube containing 50 c.c. of water, by means of a water-bath. The volume of gas collected in a measuring tube, after correction for temperature and the tension of aqueous vapour, and for the oxygen absorbed in converting cobalt oxide to peroxide (1·2 c.c. for 0·5 grm. cobalt chloride), gives the value of the sample in Gay Lussac degrees. The method is applicable to alkali hypochlorites. The specimen results show satisfactory agreement between this and the arsenious acid method. O. E. M.

Electrolytic Determination of Copper in Solutions containing Nitric Acid. E. Gilchrist and A. C. Cumming. (*Chem. News.*, 1913, **107**, 217.)—The electrolytic determination of copper in solutions containing nitric acid is inaccurate, owing to the formation of nitrous acid, with consequent incomplete deposition of the last traces of copper. It appeared probable that the addition of urea would effect an improvement, due to destruction of the nitrous acid, and experimental evidence is adduced in support of this. To prove that the gain was really due to the destruction of nitrous acid by the urea, bright copper was subjected to the action of very dilute nitrous acid solutions of different strengths, in the presence of nitric acid, both with and without urea, and in no case was more than 1 mgrm. of copper dissolved in the presence of urea, while over 200 mgrms. of copper dissolved when no urea was added. If a little urea is added towards the end of a copper deposition, the current can be stopped and the vessel washed out without any resolution of copper, provided that no unnecessary time is spent on the process. The large dilution which results from the usual siphoning method of washing is thus obviated. The best current to employ is not above 3 ampères, with a potential difference of 2·5 to 3 volts.

H. F. E. H.

Estimation of Sodium Carbonate and Copper in Mixtures of Soda Ash and Blue Vitriol by Titration with Potassium Hydroxide. H. J. F. de Vries. (*Zeitsch. anal. Chem.*, 1913, **52**, 350-357.)—Mixtures of soda ash and blue vitriol are used in agriculture, and their approximate analysis therefore assumes some importance. On account of the difficulty of titrating the strongly coloured solution which masks ordinary indicators, some analysts have been content to calculate the content of sodium carbonate from the result of a determination of carbon dioxide. But since soda ash may contain sodium bicarbonate, the results of a carbon dioxide determination may give rise to erroneous conclusions, unless controlled by a determination of total alkali. The author makes use of the first appearance of a turbidity due to basic copper sulphate as indicator. The substance is dissolved in a measured quantity of $\frac{N}{10}$ acid and the solution is boiled to expel carbon dioxide, cooled, and the excess of acid titrated with $\frac{N}{10}$ potassium hydroxide, ceasing the addition of alkali when a turbidity makes its appearance. A deduction of 0·05 c.c. is made from the consumption of alkali, this being the amount required to produce a visible turbidity in 50 c.c. of solution. If a determination of carbon dioxide is also made, the amount of sodium carbonate and bicarbonate present can be calculated.

If potassium hydroxide be added to a neutral copper sulphate solution in presence of phenolphthaleïn, there is no evidence of the presence of the indicator until the copper is almost wholly precipitated; but long before the quantity of potassium hydroxide reaches the proportion of two molecules to each atom of copper, there is a sharp colour change, easily recognised by comparison with a flask containing a similar mixture, but without phenolphthaleïn. On this observation, an empirical method has been founded for the estimation of copper in mixtures of copper salts with soda ash. After determining total alkali as described, phenolphthaleïn is added and the titration with potassium hydroxide continued until the colour of the precipitate appears to change from bright ultramarine to a violet shade. The amount of copper present is then given by the number of c.c. of $\frac{N}{10}$ alkali used, multiplied by an empirical factor. With a final volume of 45 to 70 c.c. and addition of potassium hydroxide at a rate of about 5 c.c. per minute, the factor is 0·00428. Variation in the speed of addition of the alkali has more influence on the results than reasonable variation of any of the other conditions. If the speed be only half that stated, the factor becomes 0·00434. G. C. J.

Development of Electro-analysis. R. Fresenius. (*Zeitsch. anorg. Chem.*, 1913, **81**, 4-23.)—An historical retrospect of the development of electro-analysis from the time of Wolcott Gibbs' first quantitative experiments to the present. References are given to all the original communications, including some only to be found in textbooks by their authors, which have been proved by experience to mark important advances. The tendency of recent researches to simplify apparatus on the one hand, and on the other to devise rapid methods for accomplishing difficult separations, even at the expense of some complexity of plant and procedure, is noted, and the need for and prospect of advance in both directions pointed out. G. C. J.

Determination of Hydrogen, Nitrogen, and Methane in Gas by Combustion in a Quartz Tube. Mathers and J. E. Lee. (*Chem. Engineer*, 1913, **18**, 159-162.)—The use of the Winkler-Dennis combustion pipette, even as modified by the authors, presents certain difficulties and inherent errors. In this pipette a platinum spiral is heated by means of external electrical connections, and the gases, mixed with an excess of oxygen, are burnt over mercury. If, however, the temperature be sufficiently high to insure complete combustion, the mercury is oxidised and an error due to absorption of oxygen is introduced. Probably the best method is that of Drehschmidt, in which the gas is passed through a heated platinum capillary tube. This apparatus, however, rapidly deteriorates. The authors therefore propose to substitute for the platinum capillary a quartz tube charged with pieces of scrap platinum. The quartz tube used was 30·5 cm. long, 7·25 mm. outside diameter, and 3·38 mm. inside. Its capacity was 3·317 c.c. The platinum scrap was prepared by cutting up platinum wire as short as possible, flattening the fragments, and packing them in the tube with pieces of platinum gauze at each end. The charge weighed 11·189 grms., with a volume of 0·522 c.c. A mercury pipette containing the gas and oxygen for combustion was connected to one end of the quartz tube by a capillary tube and rubber connections. A mercury burette adapted to receive the gas was connected to the other end. The burette was surrounded with a water jacket, the water of which could be circulated and mixed by raising and lowering a bottle connected with the jacket, thus preventing unequal temperatures between the top and bottom of the burette. The quartz tube was heated with a Bunsen burner with flattened flame, an asbestos board being suspended about 5 mm. above the tube to restrict radiation. The initial temperature of the gas in the pipette having been noted, the quartz tube was heated up, the air being displaced into the burette. The gas in the pipette was then slowly passed through the tube over glowing platinum into the burette, then back into the pipette, and finally again into the burette. The quartz tube was rapidly cooled, the cock connecting it to the burette was closed, and the temperature equalised in the water-jacket. After reading the volume of gas in the burette, it was corrected for variation from the initial temperature. For hydrogen, this procedure is sufficient to give accurate results. For hydrocarbons, the carbon dioxide in the burette must be absorbed and the volume thus found must be corrected for the quantity of carbon dioxide remaining in the volume of the quartz tube unoccupied by platinum. For the estimation of nitrogen, the gas must be mixed with an excess of oxygen, the nitrogen in which has been accurately determined; after combustion, the carbon dioxide and the excess of oxygen are absorbed in the usual reagents, and the residue then consists of the nitrogen in the gas *plus* the nitrogen introduced with the oxygen. J. F. B.

Volumetric Estimation of Hypophosphorous Acid and Hypophosphites. L. Marino and A. Pellegrini. (*Gazz. Chim. Ital.*, 1913, **43**, 494-497.)—A measured quantity of a dilute solution of the hypophosphite is boiled and treated with a solution of permanganate until permanently pink, after which the liquid is acidified with sulphuric acid (1 : 3) and the excess of permanganate titrated in the usual way. The permanganate solution is prepared by dissolving 6 grms. of potassium permanganate in about 800 c.c. of water, adding 40 grms. of potassium carbonate and 0·4 grm. of potassium hydroxide and diluting the liquid to a litre. After standing for a day it is filtered through asbestos and standardised upon $\frac{N}{5}$ oxalic acid solution (12·6 grms. of oxalic acid and 36 grms. of sulphuric acid (1 : 3) per litre). If a mixture of hypophosphorous and phosphorous acids be boiled with a sulphuric acid solution of a bismuth salt, a bismuthate of the metal is deposited, and the hypophosphorous acid is oxidised to phosphorous acid, which may then be estimated in the filtrate by the permanganate method described above. The results are only approximately correct, however, owing to the oxidation of the hypophosphorous acid by the bismuth being not quite complete. C. A. M.

Rapid Method of Estimating Lead Peroxide in Minium. C. Finzi and E. Rappuzzi. (*Zeitsch. anal. Chem.*, 1913, **52**, 358-367.)—The substance (1 grm.) is introduced, together with 30 to 40 c.c. of water, into a 200 to 250 c.c. flask furnished with a two-hole stopper—one hole accommodating a tap funnel, the other a gas exit tube. The gas exit tube is connected by thick-walled rubber-tubing to a Schiff's nitrometer. The water in the flask is boiled to expel air, a little water introduced through the tap funnel to clear its tube of air, and boiling continued for a few minutes, after which the rubber exit tube is clipped and the contents of the flask allowed to cool somewhat. About 20 c.c. of a solution containing 12 per cent. of hydrazine acetate and 10 per cent. of acetic acid is next run in through the tap funnel, connection is re-established with the nitrometer, and the solution is warmed and finally boiled until all the nitrogen has been driven over into the nitrometer. $N_2 = 2PbO_2$. Hydrazine sulphate is not a convenient reagent owing to its slight solubility, the reaction being very slow in presence of much water. Moreover, the lead sulphate formed tends to protect the minium from reduction. A concentrated solution of hydrazine acetate may be prepared by treating the finely powdered sulphate with excess of a concentrated solution of barium acetate, filtering off barium sulphate and diluting and acidifying the filtrate to the strength named above.

The results obtained by the method are closely concordant, but are about 7 per cent. higher than those obtained by Topf's (iodimetric) method. G. C. J.

Estimation of Manganese in Soil. M. J. Stritar. (*Zeitsch. anal. Chem.*, 1913, **52**, 337-345.)—For the estimation of manganese in soils, the bismuthate method is recommended. Small quantities of chloride (up to 0·01 grm. chlorine) are without influence on the results, provided ferric salts are absent, but this is rarely the case in an acid extract of a soil. From acid extracts of soils containing less than 1 per cent. of soluble calcium salts, hydrochloric acid is easily expelled by evaporation with sulphuric acid. But in the more common cases where the extract contains considerable quantities of calcium salts, this method is not recommended. In such cases the solution is evaporated twice or more in some cases with nitric acid; any chlorine remaining is precipitated by addition of silver nitrate, and the excess of silver precipitated by addition of the smallest possible excess of thiocyanate, the ferric salts naturally present or some added iron alum serving as indicator. The mixture is made up to known bulk, filtered through a dry paper, and an aliquot portion of the filtrate is mixed with sufficient nitric acid, treated in the cold with excess of sodium bismuthate or bismuth peroxide, filtered through asbestos, quickly decolourised by addition of a measured volume of a standard solution of hydrogen peroxide, and the excess of the latter titrated with permanganate as usual. G. C. J.

Some Quantitative Separations of Neodymium. T. O. Smith and C. James. (*J. Amer. Chem. Soc.*, 1913, **35**, 563-566.)—During the separation of the crude rare earths as oxalates from acid solutions of minerals, it has been observed that varying amounts of other elements accompany the precipitate. The experiments described in this paper were conducted with a view to determine the magnitude of the error involved in the separation of neodymium from titanium, glucinum, uranium, barium, and zirconium. When a slight excess of oxalic acid is added to a boiling solution containing 0·2 grm. of neodymia and an equal or greater amount of the oxides of glucinum and uranium, the separation is quantitative, the maximum error observed being ±0·2 mgrm. In presence of barium chloride, the recovery of neodymia was always about 0·6 mgrm. low. Since zirconium oxalate is insoluble in water but soluble in oxalic acid, the latter must be added in considerable excess to effect a satisfactory separation of zirconia and neodymia; the neodymia is always overestimated by about 0·8 per cent. It is suggested that the separation would be sharper if the solution to be analysed were poured into excess of a boiling solution of oxalic acid, the method adopted in the experiments with titanium. Of these ten experiments, seven gave results within 0·1 mgrm. of that indicated by theory, but the remaining experiments show errors of −0·6, −2·2, and −3·1 mgrms. on a weighing of 0·14 grm. G. C. J.

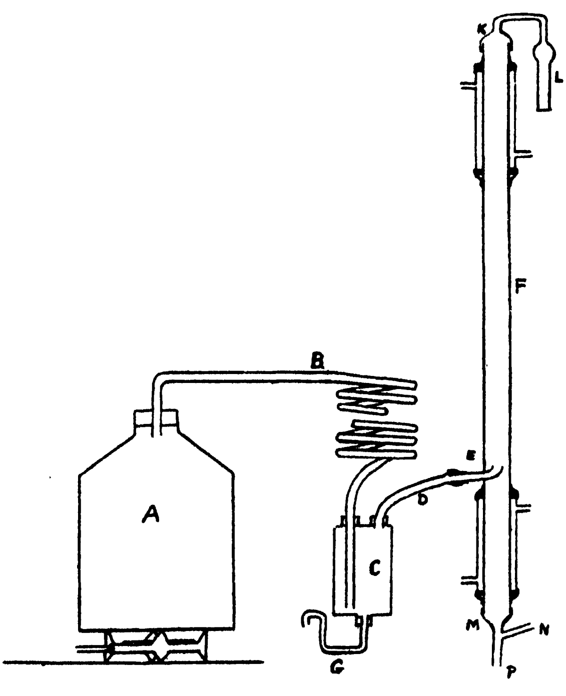
Estimation of Ozone and Hydrogen Peroxide. V. Rothmund and A. Burgstaller. (*Monatsh.*, 1913, **34**, 693-704.)—Hydrogen peroxide can be determined iodimetrically if molybdic acid is employed as catalyst. Molybdic acid is 100 times as effective as iron salts, and has the further advantage that it appears to have no accelerating effect on the reaction between potassium iodide and atmospheric oxygen. As the reaction between hydrogen peroxide and potassium iodide is very slow except in strongly acid solution, the attempt was made to estimate ozone and hydrogen peroxide in presence of each other by taking advantage of the fact that ozone reacts quantitatively with iodine in neutral or alkaline solution. After treatment with faintly alkaline or neutral iodide, the solution was made faintly acid, the iodine titrated with thiosulphate, and the solution then treated with molybdic acid, a large amount of sulphuric acid and more iodide, and the iodine liberated calculated to hydrogen peroxide. The results both for ozone and hydrogen peroxide were 10 to 20 per cent. low, and the probable causes of error are discussed in the paper. The separate estimation of ozone and hydrogen peroxide when occurring in solution together can be accomplished as follows: The solution is made faintly acid (about $\frac{N}{100}$), cooled to 0° C., and about 1 per cent. of potassium bromide is added, then at least as much potassium iodide as corresponds to the ozone, and the solution is titrated with $\frac{N}{100}$ thiosulphate. $2I = O_3$. More iodide is now added, as well as 1 c.c. of $\frac{N}{10}$ ammonium molybdate and 15 c.c. of dilute (1 : 5) sulphuric acid. After five minutes the freshly liberated iodine is titrated with thiosulphate. $2I = H_2O_2$. The process is necessarily conducted in apparatus which prevents the escape of bromine vapour. Suitable apparatus is figured in the text. The maximum error for ozone is −0·5 per cent., and for hydrogen peroxide +0·4 per cent. G. C. J.

Estimation of Phosphoric Acid. L. Moeser and G. Frank. (*Zeitsch. anal. Chem.*, 1913, **52**, 346-349.)—The following method is recommended for the estimation of phosphoric acid in mineral phosphates. It depends on the separation of all the bases as sulphates which are insoluble in strong alcohol, whereas phosphoric acid is readily soluble in that solvent. The substance is treated with about 5 c.c. of sulphuric acid, which is kept near its boiling-point for from ten to fifty minutes according to the nature of the substance. To the cooled mixture, about 30 to 40 c.c. of 95 per cent. alcohol is added, together with 2 c.c. of 10 per cent. alcoholic potash. The purpose of the potash is to give a coarsely crystalline precipitate of potassium sulphate, which carries down the finely-divided sulphates of calcium, iron, and aluminium, and assists filtration. The mixture is stirred, allowed to cool again, and then filtered through a paper previously wetted with alcohol. The residue and paper are washed four or five times with 95 per cent. alcohol, and the filtrate and washings are diluted with an equal bulk of water, made alkaline with ammonia, heated nearly to boiling, and the phosphoric acid precipitated by means of magnesia mixture. The highest and lowest results on pure tricalcic phosphate were 61·32 and 61·08 per cent. PO_4 (theory = 61·21). Four series of experiments on natural mineral phosphates show about the same extreme divergence between the results of duplicate determinations, all the results being a little higher (about 0·1 per cent.) than those obtained by the molybdate method. It is said that large quantities of manganese, such as may occur in basic slag, interfere, but no example is given to show the magnitude of the error. The small quantities of manganese present in some natural phosphorites are shown to have no measurable influence on the accuracy of the results. G. C. J.

Radiation of Uranium Solutions and Estimation of Uranium by Measurement of Radio-activity. L. Michiels. (*Zeitsch. anorg. Chem.*, 1913, **81**, 49-60.) —In the absence of radium, uranium in solution may be estimated with fair accuracy by observation of the rate at which the solution discharges a suitably designed gold-leaf electroscope. Such an instrument is figured in the paper. The solution is contained in a shallow glass dish, about 8 cm. in diameter, which is placed 3·5 cm. below the lowest point of the leaves of the electroscope. The time of discharge is independent of the depth of the layer of solution, provided this exceeds 5 mm., and depends only on the area of the dish, the concentration of the solution, and the normal leak of the instrument. With the same dish, the reciprocal of the time of discharge *less* the reciprocal of the time of equal diminution in charge in a control experiment with water is very nearly proportional to the concentration of uranium, and exactly proportional to it if the control and other solutions are adjusted to equal density by addition of ferric chloride or some other salt without measurable radio-activity.

A direct reading-form of apparatus is also described. This consists of a quadrant electrometer, of which one pair of quadrants is earthed and the other pair is connected to one plate, A, of a condenser, the second plate, B, of which is raised to a definite potential. The solution to be tested, contained in a shallow dish as described, is placed between the plates of the condenser. Finally, the pair of quadrants which are connected to the condenser are put in connection with the earth, but with a very high resistance, R (*e.g.*, a tube of xylene), interposed. In consequence of the ionisation induced by the uranium solution, an electrical current passes through the condenser, the plate A and the quadrants connected to it receive a charge, and the needle of the electrometer is deflected more and more. But since the pair of quadrants connected to the condenser is earthed through a high resistance of the order of magnitude of an air gap, a part of their charge will leak away through that resistance. The current leaking through the resistance is a function of the potential of the charged quadrants, and increases with that potential in accordance with Ohm's law; consequently a moment must arrive when the current leaking through the resistance equals the current in the condenser. At this moment the needle of the electrometer comes to rest. The greater the current passing through the condenser, the greater the potential necessary to drive an equal current through R. The potential is thus proportional to the current which, under standard conditions, is proportional to the concentration of the uranium solution. For every concentration of uranium, therefore, there is a definite position of equilibrium for the needle of the electrometer, which can be calibrated to give direct readings of uranium concentration (*cf.* ANALYST, 1913, 231). G. C. J.

Preparation of Conductivity Water. R. Bourdillon. (*J. Chem. Soc.*, 1913, **103**, 791-795.)—Work on the conductivity of dilute solutions has mostly been done with water possessing an electrical conductivity of at least 0·7 gemmho ($0{\cdot}7 \times 10^{-6}$ reciprocal ohms), requiring considerable correction and leading to anomalous results. The author describes a still which will deliver 7 to 8 litres of water of conductivity below 0·2 gemmho in a single distillation from Oxford tap-water. The boiler *A* is of copper, and holds about 13 litres. It is closed with a rubber cork, through which passes a copper tube, *B* (1·3 cm. external diameter, 8·5 metres long), coiled into a spiral of nineteen turns (thus forming an efficient spray trap), with its lower end fixed by a rubber cork in a copper cylinder, *C*. *B* and *C* are jacketed with cotton waste and boiler felt, about one-fifth of the steam is condensed here, and escapes through the glass tube *G*, bent as shown to prevent loss of steam. The uncondensed steam passes on through *D* into the condenser *F*, which is a vertical tin tube (2·5 cm. by 145 cm.), with a narrow side tube, *E*, soldered in with pure tin 45 cm. from the bottom. *F* is surrounded near the top and bottom by glass tubes, through which



cold water circulates, and is closed at the top by the glass cap, *K*, affixed by a rubber ring, and fitted with a soda-lime tube, *L*, and at the bottom by a glass cap, *M*, joined by rubber tubing at *P* to the receiver of the distilled water, and carrying a side tube, *N*, through which is introduced a continuous current of purified air (about 3 litres per hour), passing out at *L*. The air is purified by being passed through glass spirals containing concentrated sulphuric acid and potassium hydroxide solution, followed by a long soda-lime tube. The boiler is filled with tap-water, to which has been added about 5 grms. of potassium hydrogen sulphate. All connections must be kept perfectly air-tight, any leak causing a large rise in the conductivity of the water. If the distillation is carried on fairly fast (1·2 litres per hour), the conductivity of the water falls rapidly, and remains at 0·13 gemmho after one litre has been collected. Distillation should be stopped while 2 or 3 litres remain in the boiler to prevent incrustation with calcium sulphate. Similar results are obtained if phosphoric acid is used in place of the acid sulphate, but if no chemicals are used a large quantity of mediocre water is obtained, which only falls to 0·17 gemmho after four and a half hours' distillation. Very slightly better results are obtainable by starting with distilled water in place of tap-water. Tap-water, acid potassium sulphate, and no air-current result in obtaining a water which never falls below 0·25 gemmho. A condenser of only 46 cm. long proved unsatisfactory. Nothing is gained by using such low conductivity water in a conductivity cell which is not air-tight; and on storing such water in 3-litre Jena glass flasks, fitted with siphon outlet tubes, and with air inlets protected by soda-lime tubes, the conductivity rose from 0·12 gemmho to 0·2 in two or three days, and to 0·45 gemmho in a few weeks. It will keep for a month or two without exceeding 0·5 gemmho. (*Cf.* W. R. Bousfield, ANALYST, 1912, **37**, 474.)

H. F. E. H.

Estimation of Water formed on Burning Substances containing Nitrogen and Sulphur in a Calorimetric Bomb. O. Rau. (*Zeitsch. anorg. Chem.*, 1913, **81**, 116-121.)—The results obtained by the use of the bomb calorimeter include the latent heat of the steam, which is almost wholly condensed to liquid water. To calculate the lower heat value, corresponding to the production of uncondensed steam, a tolerably accurate moisture determination is necessary. The requisite degree of accuracy may be deduced from the facts that coal on combustion yields some 40 per cent. of water, the latent heat of which approximates to 3 per cent. of the total heat value of the coal, and that for the best calorimetric work an accuracy of 0·2 per cent. can be claimed. The least troublesome method of estimating the moisture is to combine this determination with the calorimetric experiment, but the nitric acid which may be derived from nitrogen in the compressed "oxygen," and the nitric and sulphuric acids resulting from the combustion of substances containing nitrogen and oxygen, complicate matters, the nitric acid tending to be absorbed and weighed as water, the sulphuric acid acting in a contrary sense by holding back moisture. Several proposals to effect the estimation with sufficient exactness are discussed and shown to be less satisfactory than the following method which is recommended. A few decigrms. of anhydrous sodium carbonate are weighed out into the bomb before the experiment, together with 20 to 30 porcelain shot. When the thermometric readings are completed, the bomb is rolled on its side, so that the sodium carbonate solution is spread by the shot all over the inner surface, and the gases are allowed to discharge slowly through tared absorption tubes containing concentrated sulphuric acid. The bomb is then heated to 110° C. in a paraffin-bath, and a current of dry air drawn through it and the absorption tubes. Moisture may be estimated in this way with an error which in no case introduces an error as great as 0·1 per cent. into the corrected heat value of the fuel. A weighed amount of sodium carbonate being taken, the determination of nitrogen and sulphur in the residue in the bomb by titration presents no difficulty. Since under ordinary conditions of combustion the sulphur in fuel is only oxidised to sulphur dioxide, and the nitrogen not at all, whereas in the bomb they are converted into sodium sulphate and nitrate, and since also the excess of sodium carbonate is converted with liberation of heat into bicarbonate, which dissolves with absorption of heat so far as the water present is sufficient for its solution, it is shown that the results of the calorimetric experiment need correction by the subtraction of

$$2020\ S + 650\ N + 160\ A - 4\ W \text{ calories}$$

where S and N represent the weight in grms. of sulphur and nitrogen oxidised, A the weight in grms. of sodium carbonate employed and W the weight in grms. of water formed. The last term, 4 W, representing the heat absorbed by the solution of sodium bicarbonate so far as the amount of water present suffices for this, is a quantity less than 2 calories, and therefore negligible compared with the quantity of 7,000 calories under measurement. The other terms represent quantities which together may amount to 1·5 per cent. of the heat value of the coal. G. C. J.

Estimation of Water in Volatile Products of Destructive Distillation. G. Lambris. (*Zeitsch. anorg. Chem.*, 1913, **81**, 24-39.)—The present paper is concerned with the accurate determination of moisture in unpurified coal gas, and the method described depends on the absorption of moisture, ammonia, pyridine, and phenol by phosphoric anhydride, retention of these substances by the phosphoric anhydride even at 180° C., at which temperature other constituents of the gas mixture which may have condensed on the surface of the reagent are expelled, and subsequent determination of and correction for the amount of ammonia, pyridine, and phenol absorbed. It is shown that neither ethylene nor benzene, toluene, xylene, or naphthalene interferes. The behaviour of substances of higher boiling-point than naphthalene was not investigated.

The purest phosphoric anhydride obtainable commercially is seldom free from yellow phosphorus, which gives rise to trouble if the contents of the drying-tube are simply distilled with caustic alkali to determine the ammonia and pyridine. If, however, the contents of the drying-tube are allowed to deliquesce by exposure to air, then treated with water and filtered through a hard filter, the finer particles of phosphorus are oxidised, and the larger ones withheld by the paper.

A measured volume of gas is passed through a tared U-tube containing phosphoric anhydride. A current of dry nitrogen or other indifferent gas is then led through the tube, which is heated to 180° C. to expel naphthalene, etc. The tube is then reweighed, and its contents are allowed to deliquesce as described. The solution of the residue is filtered and distilled with excess of caustic alkali, the ammonia and pyridine in the distillate being titrated with $\frac{N}{10}$ sulphuric acid, using 1 c.c. of a 0·1 per cent. solution of Congo red as indicator. The contents of the still are then acidified with sulphuric acid, and the phenol is distilled over in a current of steam, and estimated iodimetrically as follows: A measured and excessive quantity of a standard bromide-bromate solution is added to the distillate, and the excess of bromate determined by addition of potassium iodide and titration with thiosulphate. $6I = C_6H_5OH$.

In the neutralised distillate of ammonia and pyridine, the latter is estimated as follows: Ammonium sulphate (0·5 grm.) is added to the neutral solution, which is evaporated to dryness and heated to 130° C. for exactly fifteen minutes. Under these conditions nearly the whole of the pyridine is expelled, and the small amount left is exactly balanced by a slight loss of ammonia. The residue is taken up with water, the free sulphuric acid is titrated with alkali, using Congo red as indicator, and the result is calculated to pyridine, with due regard to the fact that the end-point of the titration is reached when only 94 per cent. of the pyridine is neutralised.

Congo red is chosen as indicator because when a solution of ammonia is mixed with one of pyridine and titrated with Congo red, the result is the same as though the two solutions were titrated separately. This is not the case when methyl orange is used. The colour-change with Congo red is much more gradual than with methyl orange, and the use of a control-flask containing a neutral solution of ammonium sulphate tinted with Congo red is essential. G. C. J.

Qualitative Analysis of the Zinc Group. R. E. Lee, R. H. Uhlinger, and F. O. Amon. (*J. Amer. Chem. Soc.*, 1913, **35**, 566-579.)—Experiments are described which show that the methods usually adopted for the separation of the metals of this group leave much to be desired, and the following scheme of analysis is recommended as certain to detect as much as 0·5 mgrm. of zinc, manganese, nickel, or cobalt in 30 to 40 c.c. of solution. The group reagent recommended is hydrogen sulphide gas, which is passed through the slightly ammoniacal solution. The latter should be warmed if less than 1 mgrm. of either metal has to be looked for. By the use of hydrogen sulphide gas in faintly ammoniacal solution, very little nickel passes into the filtrate, and this small amount is easily coagulated by boiling for a few minutes. Separation of zinc and manganese from nickel and cobalt by the use of dilute hydrochloric acid is unsatisfactory, as the latter metals are by no means insoluble in normal acid and zinc sulphide in presence of nickel or manganese sulphide is not readily soluble in weaker acid. The mixed sulphides are treated with 10 to 20 c.c. of dilute (1 : 12) hydrochloric acid, and if a black residue remains, a few crystals of chlorate are added, and the mixture boiled, diluted somewhat, and filtered from separated sulphur. The filtrate is evaporated to a few c.c. to expel excess of acid, and made slightly alkaline with 20 per cent. sodium hydroxide. If the precipitate is so bulky that a gelatinous mixture results, 10 to 20 c.c. of water are added. After cooling, excess of sodium peroxide is added by small additions, and the mixture is heated to boiling and filtered. The filtrate, which contains all the zinc and none of the other metals, is acidified with acetic acid and tested by passing hydrogen sulphide through the warmed solution. The precipitate consists of hydrated manganese dioxide, nickelic and cobaltic hydroxides. The object of peroxidising the mixture and of accomplishing this before any great excess of alkali hydroxide is present, is to insure complete precipitation of cobalt, cobaltous hydroxide being appreciably soluble in excess of alkali hydroxide. The mixed hydroxides, freed from zinc by washing, are dissolved in the least possible quantity of warm dilute (1 : 12) hydrochloric acid, and the solution is filtered from any filter-paper fluff, concentrated to about 2 c.c., and boiled with 5 c.c. dilute (1 : 20) nitric acid until red fumes cease to be evolved. About 10 to 20 c.c. of stronger (1 : 1) nitric acid are then added, together with a few crystals of chlorate, and boiling continued for a few minutes. The precipitate of manganese dioxide is filtered off on asbestos, supported on glass wool in a funnel, and its identity, almost beyond doubt, can be confirmed by the lead peroxide test. The filtrate is evaporated to 5 c.c. to expel excess of acid, diluted by addition of 10 to 15 c.c. water, and filtered if turbid. Sodium hydroxide is then added until the solution is neutral, or until a permanent precipitate begins to form. The solution is then divided into two portions, one of which is examined for the presence of cobalt by rendering just distinctly acid with acetic acid and warming with an equal volume of a saturated solution of potassium nitrite. The other half of the solution is treated with potassium cyanide until the precipitated cyanides just redissolve, heated for two or three minutes, filtered if necessary, made strongly alkaline with sodium hydroxide, and oxidised by adding bromine water until the solution is permanently red, when nickel, if present, is precipitated as black nickelic hydroxide. G. C. J.

APPARATUS, ETC.

Apparatus for the Precipitation of Barium Sulphate. H. G. Smith.

(*J. Ind. and Eng. Chem.*, 1913, **5**, 416.)—A special form of dropper is made by bending an ordinary ⅝-inch test-tube up at an angle of about 45° 1½ inches from the open end, drawing out the lower side of the bend, breaking off the small projection so formed to a point, and fusing the opening so that it will deliver about 80 drops per minute when lying horizontally on the top of a beaker with the opening downwards. The volume of barium chloride solution required for the precipitation is placed in this test-tube, and both tube and contents are immersed in the boiling solution, the dropping-point of the special tube being well above the level of the liquid in the beaker. H. F. E. H.

New Method for Determining the Diffusion of Dissolved Substances. L. W. Öholm.

(*Meddelanden fran K. Vetenskapsakademiens Nobelinstitut*, 1912, **2**, No. 22; through *Chem. Zentrabl.* 1913, I., 1648.)—Four hollow brass cylinders are placed one above the other, and diffusion allowed to take place from the bottom one, containing the solution, to the others, containing solvent. After diffusion the contents of the cylinders may be examined separately. The apparatus is intended for viscous solutions, such as lithium or potassium chloride in glycerol-water mixtures, which would adhere too strongly to glass. O. E. M.

Apparatus for the Exact Analysis of Flue Gas. G. A. Burrell and F. M. Seibert. (*U.S. Bureau of Mines, Technical Paper* 31.)—A modified Haldane apparatus, specially designed for the analysis of flue gases, including the estimation of the small proportions of methane and hydrogen which may be present in such gases. It is pointed out that as little as 0·1 per cent. of unburned combustible gases passing through a flue may represent a loss of 1 per cent. of the fuel, and that hydrogen and methane, as well as carbon monoxide, may be present, though their estimation is seldom attempted. This is attributed largely to the lack of any convenient apparatus. That now described makes possible a complete analysis of a flue gas in a little over half an hour. Duplicate analyses of the flue gas from a boiler operating under very poor conditions gave—hydrogen, 0·23 and 0·24; methane, 0·01 and 0·02; carbon monoxide, 0·98 and 1·02; oxygen, 9·43 and 9·45; and carbon dioxide, 9·90 and 9·91 per cent. G. C. J.

Extraction Apparatus for Solids and Liquids. H. Aron. (*Biochem. Zeitsch.*, 1913, **50**, 386-388.)—The material to be extracted is contained in a suitable vessel suspended in a wide glass tube, the lower end of which is attached to a flask, and the upper end of which is drawn out so as to allow of connection with condenser. The apparatus is claimed to be superior to usual forms, and can be obtained from the Vereingten Fabriken für Laboratoriumsbedarf in Berlin N. E. W.

Improvement in Gas Thermo-Regulators. A. Whitaker. (*Chem. News*, 1913, **107**, 242-243.)—Mercury, when used as a "cut-off" in ordinary gas regulators, offers many disadvantages, such as adherence to the glass, or failure to enter the fine orifice of the inlet tube. The author describes various improvements in the method of grinding the essential parts of the instrument, thus allowing for better fitting and smoother and more regular working. The end of the capillary should be countersunk (by grinding) as flat as is compatible with its object; when the regulator exhibits an angle of 135° the flow of gas is much facilitated, and is approximately equal from all points. The end of the inlet tube is ground internally to a cone, the edges being either sharp or slightly expanded, and brought very near to the prepared capillary. Such an instrument was found to be sufficiently quick in action to prevent any fluctuation exceeding 0·1° C. for several days in a bath with no stirring arrangement, provided a small bulb quick-responding thermometer is employed. For maximum sensitiveness the regulator should be clamped with the end of the inlet tube horizontal, coinciding with the surface of the mercury, thus giving a very sharp cut-off. Illustrations of the apparatus are given, and it is supplied by Messrs. A. Gallenkamp and Co., London. H. F. E. H.

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REVIEWS.

AN INTRODUCTION TO THE PHYSICS AND CHEMISTRY OF COLLOIDS. By EMIL HATSCHEK. London: J. and A. Churchill. 1913. Price 2s. 6d. net.

This book is intended to introduce readers who have a reasonable knowledge of physics and chemistry to the fundamental facts and methods of a branch of physical chemistry on the importance of which it is hardly necessary to insist. This is Mr. Hatschek's object as stated in the preface, and it has been attained in an admirable manner. For the chemical technologist and for the analyst, as well as for the physical chemist, the colloidal state of matter presents problems whose practical importance is scarcely exceeded by their profound scientific interest, and no one is better qualified than Mr. Hatschek to act as their exponent. The author employs such simple and well-chosen language, and the matter is so well arranged, that no chemist who reads this little work can fail to get a real grip of the fundamental principles of the subject. The investigation of the colloidal state has thrown a new light upon, and served to explain in an attractive manner, a large number of phenomena with which the chemist has long been familiar and with which he is brought into almost daily contact, and has shown that these, though apparently quite disconnected, are in fact often closely related. Therein lies one of the greatest charms of this vigorously developing branch of science and incidentally of Mr. Hatschek's little volume.
A. CHASTON CHAPMAN.

ALCOOL MÉTHYLIQUE, VINAIGRES. Par LOUIS CALVET. Paris: Ch. Béranger. 1912. Price 6 f.

In this little book the author deals with the subject of methyl alcohol, vinegar, acetic acid and its derivatives, on similar lines to those followed in the other volumes in this series of "Manuels Pratiques d'Analyses Chimiques."

A good summary is given of the principal methods of examining these products, and of estimating their chief constituents, special attention being given to those processes likely to be of use to Customs officials.

About half of the book is taken up with the text of the French fiscal and other regulations concerning these articles, and it is only occasionally that this information is likely to be of value to an English reader. With regard to the analytical portion, it is to be regretted that the author has not given any criticisms of the relative values of the various methods described. For example, Geissler's vaporimeter is recommended for the estimation of alcohol in vinegar (which by French law must not exceed 0·5 per cent.), and although this instrument is far from possessing the degree of accuracy sometimes claimed for it, no hint is given that the distillation method is superior.

In the section dealing with acetic acid a serious error may be noted. It is stated (p. 157) that glacial acetic acid contains from 96 to 97 per cent. of acetic acid, whilst on p. 160 the solidification-point of glacial acetic acid is given as +16° to 16·5° C. Now, this solidification-point corresponds to an acidity of over 99 per cent., whereas an acid of 97 per cent. would solidify below 12° C. In fact, in this country, acid of this strength would not be accepted as glacial acetic acid, whatever the case may be in France.
C. A. MITCHELL.

AUGUST, 1913. Vol. XXXVIII., No. 449.

THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

AN ELECTROCHEMICAL INDICATOR FOR OXIDISING AGENTS.

By ERIC K. RIDEAL, Ph.D., B.A., and ULICK R. EVANS, B.A.

(*Read at the Meeting, June* 4, 1913.)

It is well known that small traces of oxidising agents exert a considerable depolarising action at an electrode, where, in their absence, hydrogen or a reducing agent would be formed. At high concentrations this depolarising action depends only to a small degree on the actual value of the concentration of the oxidiser, and often causes a decrease of as much as a volt in the amount of the electrode potential difference. A depolarising action of this kind is utilised in the bichromate battery. A zinc and copper couple immersed in a simple acid solution will give an E.M.F. of less than a volt, but if a soluble bichromate be added this can be increased to two volts. The increase is not greatly affected by small changes in the concentration of the bichromate. On the other hand, if the oxidising agent is present in minute quantity, then the depolarising effect varies considerably with the amount actually present, and can be used as a means of estimating as well as detecting the oxidiser. The depolarising action does not cease to be measurable until the amount is very small. It must, however, be remembered that the depolarising action of the oxidiser in a cell furnishing any appreciable current will involve a destruction of part of the oxidiser as such. Thus the E.M.F. of any such system will depend on the rate at which it is made to produce a current.

This principle may be made clear by a description of one of its applications—namely, the detection of minute traces of free or available chlorine.

The addition of chlorine, hypochlorites, and ozone is becoming general for sterilising waters intended for drinking, domestic, and trade purposes, and for the treatment of sewage effluents. A difficulty is found in judging the right quantity to add, for the liquor to be treated always varies in its power of consuming chlorine or other oxidising agent, while the sterilising solution, whatever its source, also fluctuates in its oxidising power. Hence it is often found that on some days the water flows out of the treatment-tank without any excess of oxidiser; and is therefore incompletely sterilised; on other occasions the water may contain an excess of chlorine and chlorine products, which are highly injurious to whatever use the water may be put.

To insure the addition of the correct quantity of steriliser it would be necessary to keep a continuous check upon the amount of free oxidising agent in the water leaving the waterworks. What is essential, therefore, is an analytical method of estimating small concentrations of unabsorbed steriliser. Such a method must be accurate for the dilutions met with in practice; and, further, it is advisable that the whole procedure should be as automatic as possible.

It was found by us that the potassium iodide and starch reaction, usually employed in this connection, did not offer a practical basis for the solution of the problem; this is also true of the other chemical analytical methods, and we turned our attention to the physical and electrochemical changes produced by the presence of oxidising substances in water. We had to look for some property which water containing a powerful oxidising agent would possess, and which water containing no such agent would not possess. It was clear that any change which would be produced, for instance, in the density, colour, or refractive index of the water by the addition of a trace of an oxidising agent would be negligible compared to the fluctuation due to other causes. Nor would a measurement of the electrical conductivity be of use; for, although this value varies enormously with the salinity of the water, the addition of oxidising agents as such will have no appreciable effect. On the other hand, the depolarising action of an oxidising substance, which does not depend substantially on the value of the conductivity or on the presence of neutral, non-oxidising salts, may very well be used as a means of detecting and determining oxidising agents.

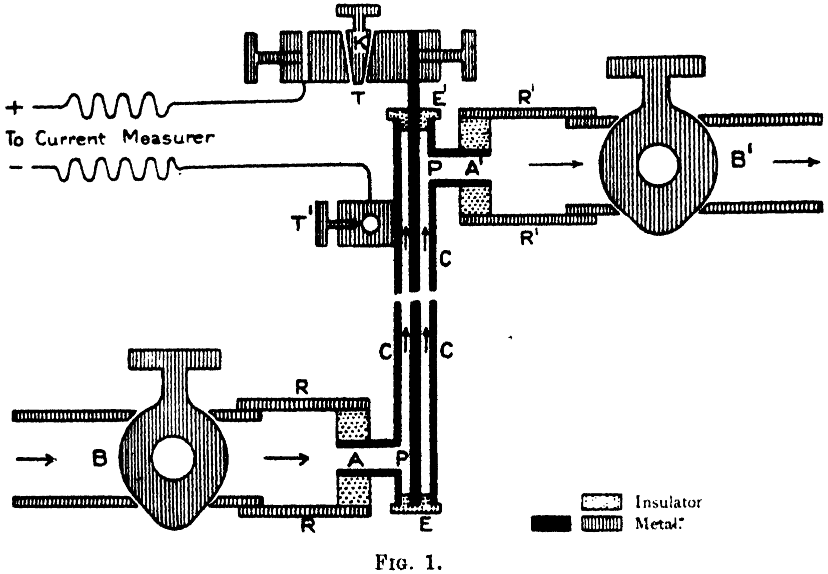
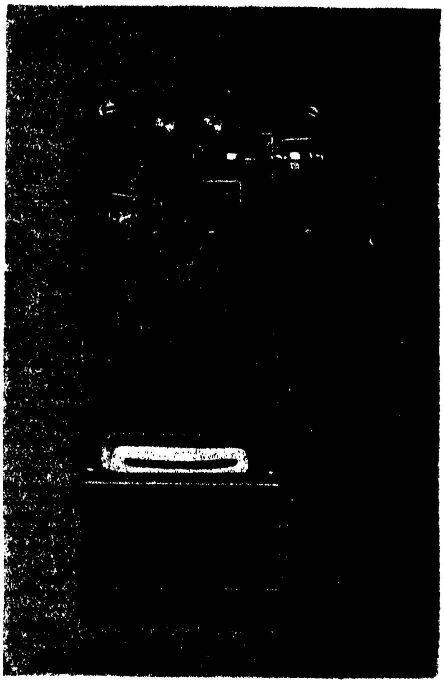


FIG. 1.

Various forms of apparatus were considered, some being experimentally tested. In the end, the form (Figs. 1 and 2) which will now be described was adopted. It consists of a copper tube, (Fig. 1) *C*, 3·5 cm. long and 4 mm. internal diameter. Within this, running axially through it, is a platinum rod, *P* (diameter 1 mm.), which is insulated at the two ends from the copper tube by the ebonite caps *E*, E^1. Liquid is allowed to enter the tube at the lower end and flow out at the top. The copper and platinum are joined to the two poles of a sensitive current-detector of high resistance; in the particular case of the instrument used in most of our experiments this had a resistance of 200 ohms, and registered ·000004 ampère per degree. If tap-water is run through the cell approximately at a constant rate, there is practically no deflection so long as oxidising agents are absent. When, however, a trace of hypochlorite or chlorine is present, a movement of the pointer over the dial occurs, and the permanent deflection produced serves as a measure of the free chlorine-content of the water. Fig. 3 shows the relation between the concentration of free chlorine added as hypochlorite and the galvanometer-reading produced by it. It will be observed that the divisions of the ammeter used in obtaining Curve *A* roughly represents parts per 10,000,000 of chlorine with the conditions observed—namely, rate of flow 230 c.c. per minute, at a temperature of 13° C. Curve *B* shows the readings obtained at higher concentrations by the use of another less sensitive ammeter of 10 ohms resistance.



It is always observed that the pointer takes a short time to reach its maximum position; and after this, if tap-water containing chlorine is being used, a slow retrogressive movement takes place, showing that the chlorine is being absorbed by the free organic matter in the water, and that it is, therefore, diminishing in amount. This has been confirmed by titration. When tap-water free from chlorine is again allowed to pass the pointer sinks at once, although it takes some minutes to reach its exact zero position. The effect of adding to the tap-water all substances likely to occur therein, in the largest quantities likely to be found in practice, has been tried, and in no case has there been any appreciable deflection produced. The additions tested include sodium chloride, sulphate, carbonate, nitrite, and nitrate, as well as ammonium salts. The effects of temperature and rate of flow have been investigated. Both these influences, as will be seen from Fig. 4, exert a considerable effect on the reading. It is, therefore, necessary to take the precaution of keeping the rate of flow sufficiently constant. In practice this is not difficult, since there will usually be the same head of water forcing the stream through the instrument. The temperature-coefficient is such that a rise of about 4 per cent. per 1° C. occurs in the reading, the head of water and other conditions being constant. There is no necessity to correct for this effect of temperature, for in warm weather the right quantity of chlorine (or other oxidising agent) which ought to be present in the water leaving a treatment station is smaller than in winter. For not only is a larger proportion of the

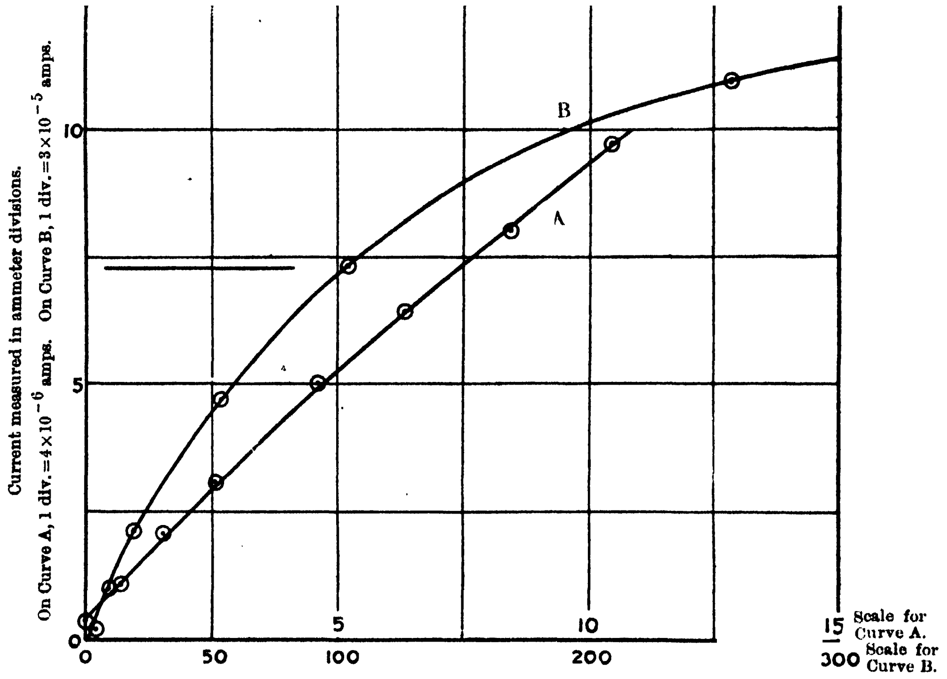


FIG. 3.—FREE CHLORINE CONCENTRATION IN PARTS PER TEN MILLION.

sterilisation done in the waterworks itself, but the oxidising agent is actually more active at the higher temperature.

Chlorine, ozone, and potassium permanganate, if present in tap-water, all cause a deflection in the apparatus. To the latter, as in the case of chlorine, the instrument is very sensitive. To ozone it is also extremely sensitive, and actually affords the best means of detecting and estimating that oxidiser, which has only a slow action on potassium iodide at the concentrations at which it gives a deflection of some degrees on the instrument. The weaker oxidising agents—*e.g.*, ferric chloride—cause no deflection. The appearance of a deflection on the latter may therefore be regarded as a sure proof that a powerful oxidising agent is present. On running a solution of hydrogen peroxide through the apparatus, a temporary deflection in the opposite direction is seen; this dies away gradually, and a low positive deflection appears. The phenomenon will serve to distinguish hydrogen peroxide and ozone at dilutions at which no other method will serve. The presence of mineral acids in the water causes a temporary positive jump. This initial deflection, appearing when an acidified liquor is first run through, soon diminishes; but in the case of a liquid containing a mineral acid never quite disappears. The deflection remaining is, in the case of hydrochloric acid, only about $\frac{1}{150}$ of that obtained with an equivalent amount of free chlorine. Of course, free mineral acids do not occur in the class of liquors to be tested.

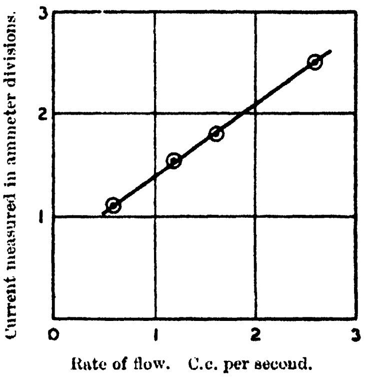


FIG. 4.

Although most reducing agents are without effect (unless they happen to be acids), phenylhydrazine hydrochloride gives a distinct negative reading, which is apparently permanent.

Finally, we may mention the fact that the current given out by the instrument is much less affected by large changes in the external resistance than would be expected from Ohm's law, assuming the E.M.F. to be constant. Although the external resistance is in both cases greater than the internal, the reading of the instrument under certain constant conditions was only changed from 7·2 to 6·7 divisions on altering the external resistance from 200 to 1,200 ohms (by the insertion of a 1,000-ohm coil).

Some of these statements may seem rather irregular, but the explanation will in most cases appear from the following discussion of the theory:

First, consider the action of the water containing a certain small amount of dissolved salts (as all waters do), but no free oxidising agent (*e.g.*, chlorine). When it flows through the tube between the copper walls and the central platinum rod a certain current will pass at first. Copper passes into solution at the one pole and turns out hydrogen at the other; thus the copper tube will be left negatively charged, for the copper assumes a positive charge on entering the ionic condition, while the platinum rod will *receive* a positive charge from the discharging hydrogen ions. But this current can only continue as long as the hydrogen concentration in and around the platinum does not exceed a certain very low value. All *ordinary* hydrogen concentrations in the platinum—if caused by extraneous agency—would actually involve an E.M.F. in the opposite direction. It is well known that hydrogen at normal pressures can remove copper from its solutions. If, however, the hydrogen concentration at the platinum be below a certain value, there is an E.M.F. tending to produce a current through the liquid from the copper to the platinum, and this E.M.F. only vanishes when the hydrogen pressure at the platinum reaches that very low critical amount depending on the electrolytic solution pressure of the copper. Now, if there were no flow through the tube and no diffusion, this concentration would soon be arrived at, and current would cease to flow. But since the water flowing over the platinum surface is continually carrying off small quantities of hydrogen in solution, the concentration is kept slightly below this value, and there is thus a small E.M.F. produced; a current therefore passes which, when a state of constancy has been set up, is exactly sufficient to replace the hydrogen carried off by the flowing liquid. With the apparatus used, which has the platinum surface small compared to that of the copper, the amount of current produced in the absence of chlorine is so small as to be barely detectable on the ammeter. It rarely exceeds 0·1 div., and can be allowed for in the zero adjustment of the instrument.

If, however, chlorine or some similar body which readily combines with hydrogen is present in the water, the case is different. The rapid attack of the chlorine keeps the concentration of hydrogen at the platinum pole permanently below the critical value, and thus there is always an E.M.F. acting from the copper to the platinum through the liquid. Therefore if these two electrodes are joined through a current measurer, a permanent current will be registered of a strength sufficient to replace the hydrogen carried off by the chlorine. It is immaterial whether the chlorine is regarded as a body which is continuously abstracting from the platinum electrode hydrogen which has already been discharged there, or whether we consider it as the active component of the electrode which attracts hydrogen ions which would not otherwise be discharged. The presence of chlorine implies that the hydrogen concentration in the platinum is kept low, although it may not reduce it, under the conditions in question, to the value demanded by the equilibrium:

$$Cl_2 + H_2 \rightleftharpoons 2HCl.$$

Passage of the liquid through the tube is required only for the renewal of the chlorine which is used up in the production of the current. The current thus becomes a measure of the amount of chlorine passing within striking distance of the platinum, and thus is not greatly dependent upon the resistance of the circuit. In other words, if the apparatus is required to furnish a current over a low resistance the E.M.F. automatically drops, whereas over a high resistance it yields nearly as great a current, but at a higher pressure.*

The reactions occurring at the two electrodes may be expressed by the following equations:

I. In Absence of Chlorine.

(*a*) At the copper pole the copper assumes the ionic condition:

$$Cu \longrightarrow Cu^{++} + 2\ominus.$$

(*b*) At the platinum pole hydrogen leaves the ionic condition. In solutions originally neutral this will clearly leave an excess of OH ions, and so confer alkalinity upon the solution:

$$2H^{+} \longrightarrow H_2 + 2\oplus.$$

As a whole, these equations can be summed up as:

$$Cu + 2H^{+} \longrightarrow Cu^{++} + H_2 + 2\oplus + 2\ominus\ ;$$

or,

$$Cu + \underbrace{2H^{+} + 2OH^{-}}_{\text{Water.}} \longrightarrow Cu^{+} + 2OH^{-} + H_2 + 2\oplus + 2\ominus,$$

an equation which involves the absorption of heat.

II. When Chlorine is Present at the Platinum Surface.

(*a*) At the copper pole the copper enters the ionic state as before, leaving the electrode negatively charged:

$$Cu \longrightarrow Cu^{++} + 2\ominus \ .\ .\ .\ .\ .\ A.$$

(*b*) At the platinum pole an equivalent amount of hydrogen ions are discharged:

$$2H^{+} \longrightarrow H_2 + 2\oplus \ .\ .\ .\ .\ .\ .\ (b)\ 1,$$

which hydrogen is to a *large extent* taken up, as it is formed, by the chlorine:

$$H_2 + Cl_2 \rightleftharpoons \underbrace{2H^{+} + 2Cl^{-}}_{\text{Hydrochloric Acid.}} \ .\ .\ .\ .\ (b)\ 2.$$

When the chlorine is present in comparatively large amount this latter equation becomes practically complete, and we can now regard the platinum-pole equation as the sum of (*b*) 1 and (*b*) 2, thus:

$$2H^{+} + Cl_2 \longrightarrow \underbrace{2H^{+} + 2Cl^{-}}_{\text{Hydrochloric Acid.}} + 2\oplus \ .\ .\ .\ B,$$

which result is identical with that obtained by regarding the platinum pole as a chlorine electrode. It will be noticed that no excess of OH^- is left behind in *this* case.

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* The comparative independence of the current upon the total resistance is of no practical value. In any case, the external resistance would be kept constant, and would be made great enough to render negligible any changes in the internal resistance due to fluctuations in the conductivity of the water used. However, the case of a chemical cell which approximates more closely to a *constant current generator* than to a constant E.M.F. generator may be of interest. The case is an exaggeration of the "polarisation" of inefficient batteries, which drop their true E.M.F. when made to discharge too quickly.

By adding A and B together we get the equation for the total reaction:

$$Cu + 2H^+ + Cl_2 \longrightarrow Cu^{++} + 2H^+ + 2Cl^- + 2\oplus + 2\ominus;$$

or omitting $2H^+$:

$$Cu + Cl_2 \longrightarrow Cu^{++} + 2Cl^- + 2\oplus + 2\ominus.$$

Since the equation, $Cu + Cl_2 \longrightarrow Cu^{++} + 2Cl^-$, represents an exothermic reaction, the source of the electrical energy needed to produce the current is largely furnished without any cooling to the liquid.

Disturbances undoubtedly may occur, due to the fact that chlorine is present at the copper pole also, and may dissolve copper chemically; it is possible, therefore, that a copper chloride concentration cell is thus set up. This effect, however, will undoubtedly be small compared to that of the main electro-chemical reaction.

Neglecting disturbances of this kind, one would imagine that the E.M.F. obtained by the cell discharging over a very high external resistance would be proportionate to

$$a + \log C_{Cl},$$

where a is a constant, and C_{Cl} is the chlorine concentration. Thus it will be almost independent of the free chlorine concentration at high concentrations of the latter. This is confirmed by experiment. The current-concentration curve becomes asymptotic at high concentrations of chlorine.

While elemental chlorine does not cause alkalinity during its depolarising action, oxidisers like sodium hypochlorite, ozone, and permanganate, presumably leave the liquid slightly alkaline.

What determines the sensitiveness of the indicator to different reagents is, however, the speed and completeness with which they reduce the concentration of hydrogen in the platinum.

The action of acids can now be readily understood. When neutral liquors have been running through the instrument for a short time, a state of constancy is arrived at in which the hydrogen contained in the platinum is practically in equilibrium with the hydrion of the liquid (it differs from the equilibrium amount only on account of the very small rate of removal due to the flow of the liquid, and replacement by the current produced). If, however, an acidified liquid with a vastly increased hydrion concentration be caused to run in, then the equilibrium relation will no longer hold; a temporary current will pass, which will tend to raise the hydrogen-concentration in the platinum. As the concentration of liquid hydrogen in the platinum approaches a value which could balance the hydrion concentration of the liquid, the current will sink; but it will never reach the minute value obtained for neutral solutions, since at its higher absolute concentrations in the platinum, the amount of hydrogen carried off by the flowing liquid will be increased. Thus acids give a high temporary, followed by a low permanent, deflection.

Again, after water free from oxidising agents or acids has been passing through the tube for some time, and has brought the hydrogen concentration in the platinum to the constant value, and then water containing an oxidiser is run through the tube, it is always possible that the oxidiser may set up a cell of the type:

$$\text{(Cu), oxidiser} \;\mid\; H_2\text{, (Pt)}.$$

The oxidiser will be reduced at the copper surface, while the hydrogen will be oxidised from the platinum and re-enter the ionic condition. This will cause a current in the opposite direction to that usually experienced, but this current can only continue until the hydrogen in the platinum has been reduced to a certain very small value, after which any E.M.F. due to this cause must cease. Then the ordinary type of change must continue, and the oxidiser will act as a depolariser on the platinum (not the copper) surface, by attacking the hydrogen forced out by the solution of the copper; this will involve a passage of a current in the normal direction. This phenomenon of *a temporary current in the wrong direction* is to be expected from oxidisers, which exert a greater power of absorbing hydrion at the copper surface than of attacking hydrogen occluded in the platinum. Undoubtedly the electrode material exerts a considerable catalytic action upon electrodic and chemical changes, and it is really impossible to say, on *a priori* grounds, as to which oxidisers, if any, will show this phenomenon. As a matter of fact, it has been observed in the case of hydrogen peroxide and sodium peroxide, and perhaps the comparative slowness with which the full positive deflection is shown in the case of hypochlorites may be due to a similar cause.

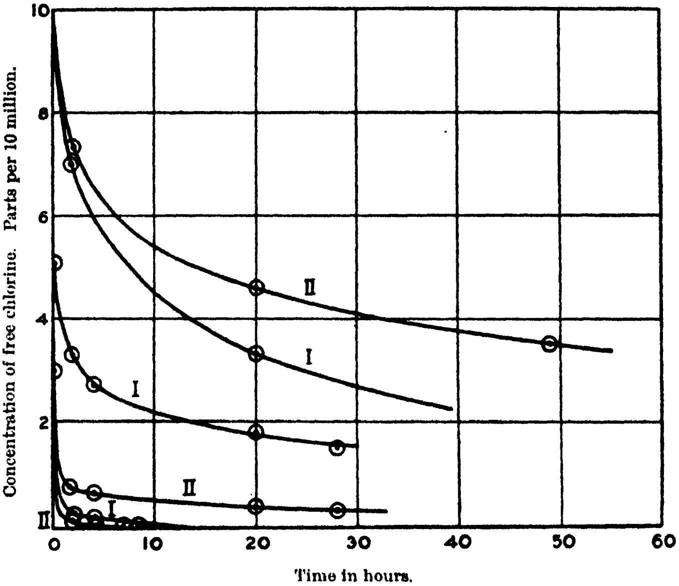

FIG. 5.

There remains to be mentioned the effect of passing reducing agents through the apparatus. Reducers sufficiently powerful to evolve hydrogen might conceivably act as a depolariser at one pole, absorbing (OH)′ ions at that pole, and causing the discharge of H+ ions at the other. As to which electrode would be the one at which hydrogen was evolved—*i.e.*, as to which would become the positive pole—prophecy is difficult. One can only say that this will depend upon the relative catalytic action of the two metals. It is possible that the reducer, instead of forcing out hydrogen at the pole at which it is not oxidised, may cause the discharge of copper ions, which are the result of some chemical attack of the copper further up the tube. At all events, the only case of a reducing agent being responsible for a current, when run through the apparatus, is that of phenylhydrazine hydrochloride; this substance sends a current in the "negative" direction (*i.e.*, from platinum to copper, through the liquid). It is possible that the explanation given in this case is incorrect, but the experiment has been repeated and the result confirmed.

As an example of the use of the "chlorometer" as a laboratory instrument, we append curves obtained by its use showing the gradual "absorption" of free chlorine by typical potable waters (Fig. 5). A certain small amount of hypochlorite of sodium was added to the water at the commencement of the experiment, and the residual amount determined from time to time by means of the apparatus. It will be seen that a great deal of the chlorine initially added is quickly taken up by the quickly oxidisable matter; after this, a slow reduction of the chlorine occurs, due to the slow oxidation of the more stable organic compounds.

DISCUSSION.

Mr. A. CHASTON CHAPMAN asked whether, when the flow-rate was increased, the readings were increased in the same proportion; in other words, with the same water, would the readings be doubled if the flow-rate were doubled? He also asked what was the nature of the influence of temperature on the readings.

Dr. LESSING asked whether the results would be affected by differences in the dimensions of the apparatus. Would it be possible that discrepant results are obtained in different sets of apparatus in which the distance between the poles is varied? It seems conceivable that a polarising current might be set up which would counteract the main reaction to a greater or lesser extent, and so influence the reading.

Dr. STEVENS asked whether the authors' investigations as to chlorine absorption had gone far enough to enable them to say what connection there was between the chlorine absorption curve and any particular characteristic of the water—the proportion of nitrites, for example.

Dr. E. RIDEAL said that an increase in the flow-rate increased the readings in considerably less proportion. Changes in temperature affected the readings to the extent of about 4 per cent. for each degree Centigrade; so it was important to keep the temperature as constant as possible. At present they had only worked with two such instruments, and had found the results to be practically the same in both cases, though for extreme accuracy exact standardisation would certainly be necessary. If the distance between the poles were unduly large, small eddy currents would be formed, and something depended upon where the water was allowed to enter and leave the instrument. They had not done much work on the subject of chlorine absorption; but different kinds of organic matter would probably be attacked by chlorine at different rates, which could be ascertained by experiment. One advantage of determining the chlorine absorption in this way appeared to be that the observations could be made at very short intervals, and an infinite gradation obtained for the different substances attacked. They had investigated the effect of some of the salts usually present in water—sodium chloride, magnesium carbonate, potassium nitrate, potassium nitrite, etc.—in larger quantities than would be present even in badly polluted water, and had found no disturbance to be caused by these.

Dr. S. Rideal remarked that chlorine was now being used for the purification of public water-supplies in all parts of the world, the latest instance he had met with being that of British Guiana, where the water in the reservoirs was being treated by adding a little bleaching-powder once a month. That seemed a crude method; but even in that case an instrument of this sort would be useful for ascertaining exactly how much available chlorine was being added, and whether the quantity was sufficient or not.

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THE ANALYSIS OF VARIOUS EAST-INDIAN TANNED HIDES.

By M. C. LAMB.

(*Read at the Meeting, June* 4, 1913.)

During the past few years the writer has often been asked by interested leather dressers whether East-Indian tanned hides were being weighted by the Indian tanner by the addition of mineral matter, or oil, more than was formerly the case. The flesh side of many East-Indian tanned hides, plastered over as they are with a mixture of a natural chalk, would certainly lead one to believe that these goods were being artificially weighted, and heavily so. With a view to ascertaining which particular tannages were the most heavily adulterated, a considerable number of analyses of samples obtained from a variety of different sources have been made. The average results of these are in the following table:

Tannage:	Bangalore.	Madras.	Ranipet.	Hyderabad.	Bombay	Coconada.	Coast.	Colombo.	Miscellaneous.
	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.
Leather Fibre	(13) 68·86	(8) 69·33	(3) 72·16	(2) 70·15	(6) 77·6	(1) 73·9	(4) 69·95	(3) 74·8	(6) 70·2
Oil and Fatty Matter ...	(16) 8·43	(3) 6·83	(5) 6·94	(3) 10·0	(8) 4·97	(3) 6·83	(4) 7·9	(4) 4·85	(8) 7·9
Water Soluble Matter .	(15) 8·6	(3) 6·76	(4) 11·3	(2) 7·5	(7) 5·55	(2) 5·9	(4) 7·55	(3) 4·86	(7) 7·4
Moisture	(13) 18·7	(3) 15·06	(3) 10·96	(2) 13·95	(6) 13·5	(1) 17·0	(4) 14·6	(3) 15·06	(6) 18·73
Mineral Matter ...	(15) 1·12	(3) 3·03	(3) 0·83	(2) 1·3	(6) 2·51	(1) 0·5	(4) 0·87	(3) 0·53	(6) 4·38

The above figures are the mean of a number of determinations of different samples. The figures in brackets indicate the number of the samples analysed.

In the case of the Bangalore tannages, which are generally considered to be one of the best of the East-Indian imported leathers, the amount of added oil and fatty matter varies from as low as 6·3 per cent. to as high as 17·9 per cent., though this latter amount is somewhat exceptional. The water soluble matter ranges from

6·7 per cent. to 10·4 per cent., though again this latter figure is higher than the average. The water soluble in every case consisted of excess tanning matter, and no sophistication with sugary matters was found to have taken place.

The Madras tannages were found to vary considerably more than others from different sources. One sample contained upwards of 7·1 per cent. mineral matter, and another no less than 13·1 per cent. of fatty matter.

The amounts of oil and fatty matter found in the Bombay samples range from as low as 2·3 per cent. to 7·2 per cent. The mineral matter, which in three cases was as low as that found in the Bangalore tannages, was in two cases very much higher, these being cuttings from goods somewhat heavily plastered, and in some cases the water soluble matter was particularly low, showing the almost entire absence of loading by over-tanning. The leather fibre figure is the highest average of any of the various tannages analysed. Owing to the limited amount of the Coconada samples available for the purpose it was only possible, except in one instance, to determine the oil and fatty matter present; this varied in the three samples from 3·8 per cent. to 10·9 per cent.

The Coast tannages are additional to the Madras tannages, and the results obtained are practically confirmatory of those obtained on the Madras samples.

The Colombo and Bombay tannages give the largest percentage of leather fibre; the amount of mineral matter is particularly low, and in two cases the amount of fatty matter is not more than would be found in a leather that had been simply oiled over for the purpose of protecting the grain surface whilst drying, and not with the idea of materially increasing its weight.

Three samples of up-country tannages were analysed for oil and fatty matter, and gave results as follows:

A.	B.	C.	Average.
5·5 per cent.	9·6 per cent.	4·7 per cent.	6·6 per cent.

Two samples of tanned buffalo gave an average leather fibre 71·3 per cent., oil and fatty matter 7·6 per cent., moisture 13·7 per cent., water soluble matter 6·9 per cent. The ash and mineral matter found was 0·9 per cent.

It would appear from the above analyses that adulteration of Indian tannages by the use of a large excess of oil and fatty matter, by use of mineral weighting matters, or an excess of tanning matter, is not nearly so common as one has been led to believe.

Whilst in many instances the amount of oil and fatty matter found is more than necessary to assist in obtaining a good coloured leather on drying by providing a film of oil to the grain side, except in a few cases the gain of weight obtained by the addition of oil is not so great as one would have expected by a visual examination. The analyses compare very favourably indeed with the home-tanned calf and dressing hides, and prove that adulteration is not general amongst this class of leather.

In conclusion, I would like to record my indebtedness to Mr. Charles Furneaux for carrying out the detailed analyses, and to Messrs. Flack, Chandler and Co., Culverwell, Brooks and Co., Dyster, Nalder and Co., Samuel Baprow and Bros., Scriven Bros., Malcolm Inglis, and others who have supplied authentic samples.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Analysis of Amyl Acetates and Collodions. N. Chercheffsky. (*Les Matières Grasses*, 1913, **6**, 3103-3110.)—The so-called pure amyl acetate of commerce consists of a mixture of amyl acetate and amyl alcohol. Its purity may be ascertained by a determination of its principal physical constants, and the proportion of amyl acetate may be calculated from the saponification value :

	Sp. Gr. at 15° C.	Boiling-Point. °C.	$[N]_D$ 15° C.	Oleorefractometer Reading at 15° C.	Saponification Value.
Amyl alcohol (iso-) ...	0·820	129–131	1·4100	+29	—
Amyl acetate (iso-) ...	0·875	139	1·4035	0	431
Ethyl acetate ...	0·794	78·4	1·3640	-170	—
Propyl acetate (normal)	0·807	98	1·3890	-60	—
Acetone	0·792	56·3	1·3640	-170	—
Benzene	0·887	80·5	1·5050	+420	—
White spirit	0·777	150–170	1·4365	+135	—

In commercial amyl acetate derived from fusel oils the impurities may include ethyl acetate, normal propyl acetate, iso-butyl acetate, and their corresponding alcohols. Their presence is indicated by the lævo-oleorefractometer readings obtained with the products of the fractional distillation of the sample into ten parts. The boiling-points of these fractions will also differ materially from those of the corresponding fractions of pure amyl acetate. The fraudulent addition of larger quantities of ethyl acetate may be detected by boiling 5 c.c. of the sample for thirty minutes beneath a reflux condenser with 25 c.c. of a 15 per cent. solution of potassium hydroxide in amyl alcohol, distilling the saponified product, and collecting the first 1 to 1·5 c.c. of the distillate. This fraction is shaken with water, and the aqueous layer tested with potassium carbonate and iodine. A distinct iodoform reaction is obtained if the sample contained 1 per cent. of ethyl acetate. For a quantitative estimation, 50 grms. of the sample are saponified with a solution of 25 to 30 grms. of potassium hydroxide in about 50 grms. of amyl alcohol, the product distilled, and about 50 grms. of the distillate shaken in a separating funnel with 100 c.c. of calcium chloride solution of sp. gr. 1·250 and 30 c.c. of pure cymene. The lower layer is drawn off, the shaking twice repeated, each time with 50 c.c. of fresh calcium chloride solution, and the united saline layers distilled. The first 50 c.c. of the distillate are filtered, the sp. gr. determined, and the corresponding percentage volume of ethyl alcohol thus obtained. The result multiplied by 1·508 gives the percentage of ethyl acetate (by weight) in the sample of amyl acetate. The method gives good results provided large amounts of propyl and butyl acetates are not present. Acetone may be detected and approximately estimated by the following thermometric method: Ten c.c. of the sample are exactly neutralised with sodium hydroxide, and treated in a Dewar's vacuum-tube with 5 c.c. of sodium bisulphite solution (sp. gr. 1·320), and the rise in temperature noted. The number of degrees C. corresponds approximately to the percentage of acetone (by volume), and satisfactory results are thus obtained for amounts ranging from 5 to 20 per cent. Cyclic and acyclic hydrocarbons are detected by the physical properties, and especially the oleorefractometer reading. As a further test, from 2 to 3 c.c. of the sample are shaken with an equal volume of sulphuric acid (sp. gr. 1·84) in a stoppered graduated cylinder. In the presence of benzene (10 per cent.) a characteristic turbidity appears after the cylinder has stood for five to ten minutes, whilst with toluene and xylene, solution takes place without subsequent turbidity. When acyclic hydrocarbons are present, however, a supernatant layer is formed, and the amount may be measured after the separation is complete (five to ten minutes). In examining collodion, the nitrocellulose is precipitated by means of petroleum spirit boiling below 70° C., and the precipitate dried at 90° C. on a tared filter. Another portion is evaporated to dryness, and the residue weighed, the results thus obtained being usually somewhat higher than in the nitrocellulose estimation. The solvent is quantitatively distilled in a current of steam from 50 grms. of the sample, sodium chloride being placed in the receiver to prevent solution of ethyl acetate in the water. Any ethyl acetate dissolving in the saline liquid may be detected by the iodoform reaction. The separated solvent is then examined by the methods described. C. A. M.

Chloral Hydrate Test for Charlock. A. L. Winton. (*Eighth Inter. Cong. App. Chem.*, 1912, vol. 26, 409.)—Wagge (*Ber. d. Pharm. Ges.*, 1893, 153) first described the detection of charlock in mustard flour by means of chloral hydrate. Freshly made chloral solutions, which are not at all acid, fail to give the test, but do so at once if acidified, or if kept till free hydrochloric acid is liberated. The test is best performed as follows: Sixteen grms. of the chloral hydrate crystals are dissolved in 10 c.c. of water, and 1 c.c. of strong hydrochloric acid are added. About 10 mgrms. of the mustard flour are placed on a slide in the reagent, the whole carefully heated (never to boiling) for a moment, and then examined under a lens. The proportion of fragments of hulls that acquire a carmine red colour (charlock) to those that do not is then noted. H. F. E. H.

Comparative Study of the Composition of Hops grown in Different Parts of the World. H. V. Tartar and B. Pilkington. (*J. Ind. and Eng. Chem.*, 1913, **5**, 478-480.)—Representative samples of hops from different parts of Europe and America were analysed in the endeavour to correlate prices and quality, considerable variations in brewing value being set upon the crops from different localities. It is considered that the major part of the actual brewing value of hops lies in the content of soft bitter resins, and the authors consider that their results indicate that hops which hold a high position in the market are in reality inferior in composition to others which are rated lower commercially. Especially is this the case with certain European samples. Siller's method (ANALYST, 1909, **34**, 485) for the separation of the soft α and β resins was employed, and Chapman's cinchonine method for the estimation of tannin (*ibid.*, 1908, **33**, 95, and 1909, **34**, 372). Wax is the matter soluble in ether and insoluble in alcohol. The hard, total, and soft (bitter) resins were determined by the methods of Tartar and Bradley (*ibid.*, 1912, **37**, 191). The following are some of the results recorded in the paper. In all cases the samples were of the 1911 crop:

Locality.	Moisture.	Total Resin.	Total Soft Resin.	Hard Gamma Resin.	Soft Beta Resin.	Soft Alpha Resin.	Wax.	Tannin.	Seeds.	Leaves and Stems.
Washington (State)	3·90	17·55	15·64	1·91	14·85	0·79	0·37	2·02	4·86	5·00
New York, No. 1 ...	2·40	14·20	12·81	1·39	8·92	3·89	0·60	1·30	2·50	15·40
„ „ No. 3 ...	4·10	18·51	16·61	1·90	13·81	2·80	0·71	3·06	0·06	9·00
California, No. 2 ...	3·70	17·10	15·72	1·38	11·65	4·07	0·52	2·52	18·35	8·80
„ No. 4 ...	5·70	15·76	14·10	1·66	9·14	4·96	0·33	1·40	7·55	13·50
Wisconsin	4·00	15·82	8·68	7·14	7·93	0·75	—	2·40	0·22	1·08
England: Worcester	2·50	12·21	9·97	2·24	6·60	3·37	0·50	1·16	9·55	1·20
„ East Kent	2·50	11·70	10·51	1·19	7·30	3·21	0·47	1·58	7·90	0·90
„ Sussex ...	4·10	12·70	10·35	2·35	6·05	4·27	0·44	2·22	6·15	0·60
„ Farnham	4·80	12·34	11·06	1·28	6·65	4·41	0·39	1·66	12·50	0·00
Oregon, No. 1 ...	6·30	18·35	16·75	1·60	10·56	6·19	0·33	1·67	11·50	7·60
„ No. 4 ...	6·80	19·80	17·62	2·18	9·48	8·14	0·41	1·99	1·42	4·00
Saaz, No. 1... ...	6·50	16·25	14·19	2·06	7·20	6·99	0·54	—	Nil	Nil
„ No. 2... ...	8·20	13·75	12·60	1·15	7·62	4·98	0·45	—	Nil	Nil

H. F. E. H.

Estimation of Glycyrrhizin in Sweets and in Liquorice. E. Durier. (*Ann. Falsif.*, 1913, **6**, 252-255.)—The following method is recommended for the estimation of glycyrrhizin in sweets, the quantity of this liquorice constituent found being a measure of the amount of liquorice present. "Liquorice juice" contains from 8 to 25 per cent. of glycyrrhizin, and, taking the minimum quantity as a standard, sweets should yield at least 0·32 per cent. of glycyrrhizin to indicate that they contain not less than 4 per cent. of "liquorice juice," this being the minimum quantity allowed by French law in liquorice preparations. Twenty grms. of the finely divided sample are extracted for fifteen hours at the ordinary temperature with 10 per cent. ammonia; 150 c.c. of 95 per cent. alcohol are then added slowly, and, after five hours, the liquid is poured through a filter, and the precipitate is washed with about 100 c.c. of 70 per cent. alcohol. The filtrate and washings are evaporated to a syrup; this is dissolved in 50 c.c. of water, 1 c.c. of ammonia (sp. gr. 0·92) is added, and, after the lapse of thirty minutes, the glycyrrhizic acid is precipitated by the addition of 2 c.c. of hydrochloric acid. At the end of twenty-four hours the precipitate is collected on a filter, washed with small quantities of water (25 c.c. altogether), dried for thirty minutes at 100° C., then dissolved in ammonia, the solution is filtered, the filtrate evaporated to dryness, and the residue weighed after being dried for six hours at 100° C. To the weight found is added 0·023 grm. to correct for the solubility of the glycyrrhizic acid in the quantity of water used.

A similar method is used in the case of liquorice extract, but the preliminary extraction with ammonia and precipitation with alcohol must be repeated several times. W. P. S.

Freezing-Point of Milk. J. B. Henderson and L. A. Meston. (*Proc. Roy. Soc., Queensland,* 1913, **24**, 165-181.)—The authors find that the freezing-point of pure fresh milk from herds of cows in Southern Queensland never shows a greater variation than from − 0·55° C. to − 0·56° C., the mean being − 0·555° C. They are of opinion, as the result of their experience, that the freezing-point determines with accuracy the proportion of water added to any milk from a herd, and distinguishes watered rich milk from naturally poor milk. W. P. S.

Method of Distinguishing Jamaica Rum from Artificial Rum. H. Fincke. (*Zeitsch. Untersuch. Nahr. Genussm.,* 1913, **25**, 589-596.)—Estimations of the free and combined formic acid in rums afford data which are of some use in determining the character of the spirit, especially when compared in conjunction with the other analytical results. The author finds that Jamaica rum contains from 3·28 to 5·03 mgrms. of free formic acid per 100 c.c., and from 3·34 to 4·45 mgrms. of formic acid in the form of esters. Artificial rum contains from 2·68 to 26·01 mgrms. of free formic acid, and from a trace to 4·84 mgrms. of combined formic acid per 100 c.c. Whilst in the case of Jamaica rum the free and combined formic acid is present in approximately equal quantities, the relation between the amounts varies widely in the artificial spirit. W. P. S.

Composition of Port Wine. A. Kickton and R. Murdfield. (*Zeitsch. Untersuch. Nahr. Genussm.,* 1913, **25**, 625-675.)—Analyses of some hundreds of samples of port wine are recorded, the samples for the most part representing the wine produced by recognised Portuguese firms and imported into Germany. The results obtained were as follows: Alcohol, 14·5 to 17·5 per cent. by weight, or 18 to 22 per cent. by volume; total solids (including sugars), 8·0 to 11·5 per cent.; total acid, 0·3 to 0·5 per cent.; sugar-free extract, 1·5 to 2·5 per cent.; ash, 0·15 to 0·3 per cent.; glycerol, 0·5 to 0·7 per cent.; phosphoric acid, 0·015 to 0·030 per cent. As a rule, the wines contained considerably more lævulose than dextrose. The white port wines examined did not differ in their composition from that of the red wines.

W. P. S.

Estimation of Glycerol in Wines. E. Bertainchand. (*Ann. Falsif.,* 1913, **6**, 278-281.)—Fifty c.c. of the wine are neutralised with barium hydroxide solution and evaporated at a temperature of 70° C. to a volume of 10 c.c.; in the case of red wines, a small quantity of animal charcoal is added at this point. The evaporation is then continued at 60° C. until a syrup is obtained; to this are added 5 grms. of sand and, when the mixture is cold it is extracted four times with absolute alcohol, using 5 c.c. of the latter each time. The alcoholic extracts are decanted, without filtration, into a 100 c.c. flask. The residue is then extracted four times with pure ethyl acetate, 20 c.c. being used each time, and the extracts are added to the alcohol solution. The contents of the 100 c.c. flask are made up to the mark with ethyl acetate, a further 1 c.c. of the latter is added to correct for the volume of the insoluble matter, and, after shaking, the turbid solution is filtered. Eighty c.c. of the filtrate (40 c.c. of wine) are evaporated at 60° C., and the residue of glycerol is dried at this temperature to constant weight. W. P. S.

Estimation of Sulphate in Wines. C. von der Heide. (*Zeitsch. anal. Chem.*, 1913, **52**, 440-451.)—More sulphate is found in the ash of wines than is found by addition of barium chloride to the wine itself. In the author's experiments the difference could not be attributed to sulphur derived from gas used for evaporation and ignition, and was found to be due to the oxidation of sulphur dioxide and organic sulphur compounds during evaporation and ignition. The ratio of organic to sulphate sulphur may be as high as 1 : 5, and no wine is quite free from sulphite. Even when sulphate is determined without previous incineration, some of the sulphur dioxide present is oxidised to sulphate, and the author recommends previous distillation of the acidified wine in a current of carbon dioxide. This distillation may be made to serve simultaneously for the estimation of sulphite, but phosphoric acid should not be used for acidifying the wine, if the residue in the still is to serve for a sulphate determination. Some 10 c.c. of 10 per cent. hydrochloric acid is used to decompose sulphites, and no oxidation need be feared if this acid be diluted with 100 c.c. of water, heated to boiling, and the flask filled with carbon dioxide before introducing the wine. G. C. J.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Application of Dialysis and Estimation of Oxidising Power as Aids in Judging Soils. J. König, J. Hasenbaümer, and K. Glenk. (*Landw. Versuchs-Stat.*, 1913, **79-80**, 491-534; through *Chem. Zentralbl.*, 1913, I. 2001.)—The authors have followed up the work of Petermann (*Landw. Versuchs-Stat.*, **14**, 465), who first suggested the use of dialysis in the estimation of the available plant food in soil. They record the fact, established by them, that, when soils are heated, the percentage of dialysable material is increased, but their researches lead them to the conclusion that dialysis is not likely to prove of much service in the judgment of soils, as the results are too irregular and the time occupied by the experiments too long.

As the acids formed in the soil dissolve and render available as plant food the various constituents of the soil, the authors suggest that the estimation of the oxidising power of a soil, that is to say, the rate at which carbon dioxide and nitric acid are produced in it, should aid in the formation of a correct judgment as to its probable fertility. They have studied the effect of additions of dextrose to soils. Such additions increase the rate of formation of carbon dioxide and decrease the electrical conductivity of the soil. Soil which has been heated gives better yields in pot cultures than soil which has not been so treated. G. C. J.

Method for Detecting the Vitality of Seeds. S. Tashiro. (*Eighth Inter. Cong. App. Chem.*, 1912, vol. 26, 163.)—With an apparatus capable of detecting and measuring as little as one ten millionth of a grm. of carbon dioxide, the author has been enabled to show that a dry living seed liberates this gas as long as it is alive. By stimulating a living seed by bruising or crushing, a large increase in the liberation of carbonic acid was observed. H. F. E. H.

Estimation of Lactic Acid in Urine. M. Dapper. (*Biochem. Zeitsch.*, 1913, **51**, 398-407.)—Five hundred c.c. urine are concentrated at 50° to 60° C. *in vacuo* to 100 to 120 c.c., 50 per cent. phosphoric or sulphuric acid added, and extracted in a Lindt apparatus for twenty-four hours with pure ether. The ether extract, after addition of ammonia, is concentrated on the water-bath, the residue introduced into a flask, 300 c.c. dilute sulphuric acid added, and distilled. After 200 c.c. have distilled, the residue is oxidised with permanganate, distilled, and the aldehyde in distillate estimated by titration with bisulphite and iodine according to the method of V. Fürth-Charnasz (ANALYST, 1910, **35**, 445). In order to guard against possibility of urine containing substances other than lactic acid which on oxidation yield products that react with bisulphite or iodine, the author recommends the analysis of the zinc salt prepared from isolated lactic acid. E. W.

Search for Pathogenic Microbes in "Raw" River Water and in Crude Sewage. A. C. Houston. (*Ninth Research Report, Metropolitan Water Board.*)—The author suggests as an alternative title for the present report, "Where is the Typhoid Bacillus?" and answers the question as follows: "The home of the typhoid bacillus is not so much in impure water, or even in the crude sewage from a large community, as in the 'factories' of disease, as exemplified by the 'carrier' case." Owing to the discovery of typhoid "carriers," the question of accident has assumed a new and even a deadly significance. We know now that a relatively gross pollution derived from the sewage of a large community *may be* less dangerous than traces of contamination coming from a single individual of unknown health-history. If the danger unit is a carrier (and some authorities estimate that three to four out of every 1,000 persons are in this condition), his or her discharges may be 10,000 times more infectious than crude sewage volume for volume. The danger of pollution (volume for volume) would seem to rank as follows: (1) The carrier unit, the concentrator and factory of disease—the genesis of epidemics; (2) the unit person of unknown health-history, who may be in the position of (1), and is therefore, in a potential sense, placed second, but who in the great majority of cases, one would hope, is an almost negligible factor; (3) the collection of individuals on a large scale, exemplified in a contaminating sense by the sewage of a large town. Here, the dangerous "carrier" element being reduced by dilution to normal proportions, less than one typhoid bacillus per 0·001 c.c. of sewage might be anticipated to be present on the basis of the foregoing observations.

Attention is drawn to the fact that in American cities the typhoid fever death-rate compares very unfavourably with European cities, as the following selected figures show:

	Population.	Death-rate per 100,000.
Edinburgh	320,000	1·3
Dresden	550,000	2·2
Berlin	2,000,000	2·9
London	7,280,000	3·3
Vienna	2,000,000	3·8
Hamburg	950,000	4·1
Paris	2,750,000	5·6
Boston	670,000	11·3
New York	4,766,883	11·6
Philadelphia	1,549,008	17·5
Washington	331,069	23·2
Minneapolis	301,408	58·7

American sanitarians especially attribute a considerable proportion of their total typhoid fever cases to the consumption of impure water; but in the author's view the part played by water-supply in causing endemic typhoid fever is apt to be exaggerated, especially as it can be shown by calculation that the customary draught of water (half a pint) of a satisfactory, or perhaps even of an unsatisfactory, water-supply does not contain 1,000 excremental bacteria, and therefore cannot be expected to contain even a single typhoid bacillus. In cases, however, where the pollution is from the urine of a typhoid carrier, there may be no fæcal bacteria, yet an abundance of typhoid bacilli. H. F. E. H.

ORGANIC ANALYSIS.

Detection of Benzoic Acid in the Presence of Phenol and Salicylic Acid. L. Robin. (*Ann. Falsif.*, 1913, **6**, 277-278.)—The test consists in nitrating the benzoic acid and reducing the *m*-dinitrobenzoic acid by means of ammonium sulphide, an amino compound having a red colour being thus obtained. Phenol and salicylic acid also yield a red coloration; but these two substances may be destroyed by the action of permanganate in sulphuric acid solution, whilst the benzoic acid is unaffected. The ethereal solution of the benzoic acid, and possibly of phenol or salicylic acid, obtained in the usual manner from a food or other substance, is shaken with a mixture of 20 c.c. of water, 5 c.c. of alcohol, and 0·2 grm. of sodium hydrogen carbonate. The aqueous solution is separated, evaporated, the residue is taken up in a small quantity of water, and, after acidifying with sulphuric acid, the solution is heated to 80° C., and potassium permanganate solution is added drop by drop until a pink coloration is obtained. After cooling, the mixture is again extracted with ether and the salicylic acid obtained from the ethereal solution is identified by means of the test described above. W. P. S.

Characteristics and Differentiation of Native Bitumens and their Residuals. C. Richardson. (*J. Ind. and Eng. Chem.*, 1913, **5**, 262-266.)—Native bitumens differ so much in character from one another that means of differentiating between them are much to be desired, especially by those who wish to utilise them or their residuals for highway construction. The most important considerations in characterising native bitumens are the ratio of saturated to unsaturated hydrocarbons, the percentage of sulphur, and, in the case of petroleums, the proportion of this sulphur which is removed on distillation in a current of steam, and the percentage of coke (ash-free) which they yield under defined conditions. The viscous oils remaining in the still when various petroleums are distilled to 325° F. are used for fluxing solid asphalts for road making, and are referred to as fluxes. Paraffin fluxes, the residuals from paraffin petroleums, are characterised by their notable content of paraffin scale (4 to 14 per cent.), by the predominance of saturated hydrocarbons (74 to 86 per cent.), comparatively low density (0·92 to 0·94 per cent.), low sulphur content (0·4 to 0·6 per cent.), and low residual coke (2 to 4 per cent.). These paraffin fluxes will flux true asphalts, but not gilsonite or grahamite. Mexican petroleum stands in a class by itself, and the residual flux obtained from it contains only 2 per cent. paraffin scale and 40 to 50 per cent. saturated hydrocarbons, has a density exceeding 1, contains 4 per cent. of sulphur, and gives 10 per cent. residual coke. Between the typical paraffin fluxes and those derived from the true asphaltic petroleums of Trinidad and California stand a class of fluxes derived from the semi-asphaltic petroleums of Texas, which are valuable road-making material, since they flux satisfactorily such solid bitumens as gilsonite and grahamite. They are characterised by a low content of paraffin scale (0·3 to 4·0 per cent.), a somewhat lower percentage of saturated hydrocarbons (68 to 80 per cent.) than the paraffin fluxes, a higher density (0·95 to 0·97), and low sulphur and low residual coke. Fluxes derived from true asphaltic petroleums contain no paraffin scale, under 50 per cent. saturated hydrocarbons, 6 to 7 per cent. residual coke, usually much sulphur (2 to 3 per cent.), and have a density above 1. Bitumens derived from residual fluxes by treatment with sulphur or air at high temperatures have appeared on the market. They are short, or lacking in ductility and cheesy, or non-adhesive. The result of the treatment is to increase the percentage of residual coke (to 9 to 20 per cent.), reduce that of saturated hydrocarbons (to 35 to 65 per cent.), and introduce more or less sulphur, according to the process adopted. The sulphur may approximate 5 per cent., but is usually less than 2 per cent.

The solid residual pitches, used for highway construction, fall into four main classes, corresponding to their origin in Texas, California, Trinidad, and Mexico. Texas residual pitches are characterised by a small percentage of paraffin scale (0·5 to 1·2 per cent.), less than 1·25 per cent. of sulphur, and a very high percentage of residual coke (17 to 24 per cent.). Californian residual pitches are distinguished from those derived from Texas oil by the lower percentage of saturated hydrocarbons (26 to 32 per cent. against 48 to 66 per cent.), and the lower yield of residual coke (9 to 13 per cent.). They carry but a trace of paraffin scale (nil to 0·6 per cent.), and less than 1·4 per cent. of sulphur. Trinidad residual pitches are differentiated from those above described by their sulphur content (2 per cent.), and further from Californian products by their low content of saturated hydrocarbons (24 to 31 per cent.). Mexican residual pitches contain 5 to 7 per cent. of sulphur, which sharply differentiates them from the products above described. They contain 1·0 to 2·4 per cent. paraffin scale, and yield 12 to 30 per cent. of coke.

Native asphalts contain 4 to 8 per cent. of sulphur, less than 25 per cent. of saturated hydrocarbons, and no paraffin scale. The glance pitches (Egyptian and

Manjak), very hard and brittle materials, contain only 6 to 7 per cent. of saturated hydrocarbons, whilst, of the solid native bitumens other than asphalt, gilsonite contains 6 per cent. and grahamite a mere trace (0·3 per cent.). G. C. J.

Hardened Marine Animal Oils. C. Grimme. (*Chem. Rev. Fett Ind.*, 1913, **20**, 129-134.)—Two samples of hydrogenised marine animal oils, which superficially were indistinguishable from lard and tallow respectively, gave the following analytical values:

Sp. Gr. at 15° C.	Melting-Point.	Solidifying Point.	$[N]_D$ 40° C.	Acid Value.	Saponification Value.	Iodine Value (Wijs).
	°C.	°C.				
0·9271	47·2	34·9	1·4529	1·94	189·3	23·24
0·9200	38·5	31·6	1·4575	1·00	188·8	58·34

Hardened fish and marine animal oils give characteristic colour reactions, the intensity of the coloration depending upon the extent to which they have been hydrogenised. For example, the following colorations were obtained:

	HARDENED OILS.			
	Seal Oil.	Whale Oil.	Liver Oils.	Fish Oils.
Fuming nitric acid ...	Red brown.	Brown; afterwards black-brown.	Blood-red, then bluish-red to brown.	Brown.
Sulphuric acid (sp. gr. 1·65—1·70)	Red-yellow, then brown-red.	Brown, then black-brown.	Violet to violet-black.	Green, then brown to black.
Nitric and sulphuric acids (1 : 1)	Red, then brown.	Yellow, then red-brown.	Yellowish-red, onion red, and finally red-brown.	Yellow, green to brown.
Syrupy phosphoric acid ...	Brown.	Red-brown.	Red.	Red-brown.
Aqua regia	Pale yellow mass.	—	—	—
Sodium hydroxide ...	—	Red-brown.	Red.	Red-brown.
Phosphomolybdic acid (1 per cent.) added to chloroform solution of fat	Blue ring at junction of liquids.	—	—	

The colorations given by the hardened oils did not correspond to those described as characteristic of the original fish and marine animal oils. Nickel may best be detected in these fats by means of Fortini's dimethylglyoxime reagent. C. A. M.

Estimation of Carbon Tetriodide in the Presence of Iodoform. M. Lantenois. (*Comptes rend.*, 1913, **156**, 1629-1631.)—Silver nitrate (in 20 per cent. solution) reacts with iodoform giving a quantitative yield of carbon monoxide and silver iodide. Carbon tetriodide under the same conditions yields on the one hand carbon monoxide and carbon dioxide and, on the other hand, silver iodide and iodate, the two reactions apparently occurring simultaneously:

$$CI_4 + 4AgNO_3 + 2H_2O = CO_2 + 4HNO_3 + 4AgI.$$
$$3CI_4 + 12AgNO_3 + 6H_2O = 3CO + AgIO_3 + 11AgI + 12HNO_3.$$

For a gravimetric estimation of the carbon tetriodide it would therefore be necessary to reduce the iodate, but a gas-volumetric method may be based upon the fact that whatever the relative proportions of carbon monoxide and dioxide, their total volume is directly proportional to the weight of carbon tetriodide. This reaction may be used in the estimation of carbon tetriodide in the presence of iodoform, the measurement of the gas affording an exact estimation of the amount of carbon in the compound. One grm. of carbon tetriodide yields 42·9 c.c. of gas, whilst 1 grm. of iodoform yields 56·6 c.c. C. A. M.

Estimation of Organic Matter in Clays. P. Ehrenberg, C. Diebel, and H. Veckenstedt. (*Zeitsch. anal. Chem.*, 1913, **52**, 408-418.)—The authors confirm the statement of earlier workers (*e.g.*, Warington and Peake, *J. Chem. Soc.*, 1880, **37**, 617) that the organic carbon of clays cannot be determined with certainty by methods depending on moist combustion. Some clays yield the whole of their carbon as carbon dioxide when treated with chromic acid, others less than half. In no case was more than 80 per cent. of the carbon obtained as carbon dioxide when permanganate was employed as oxidising agent. The following method is recommended: From 10 to 20 grms. of the air-dried clay is treated with at least enough phosphoric acid to decompose any carbonates present. The mixture is dried, then ground up and mixed with finely divided copper oxide, and a combustion made in the usual manner. A number of clays examined by this method showed from 0·1 to 0·7 per cent. humus when the organic carbon was calculated to humus by the factor 1·7. As they are unable to find any record of clays with more than a small fraction of the 15 per cent. of humus said by Rohland (*Zeitsch. Chem. Ind. Kolloide*, 1906, **1**, 78) to be occasionally present, the authors conclude that Rohland's statement was based on some one rare specimen. They do not disagree with Rohland's view that the organic matter of clays may be one of the factors which determines their plasticity and other physical properties, and agree that even such small amounts as they found may conceivably exercise a profound influence. But since their samples included the whole range of clays used in a large works, they conclude that the maximum effect of the organic matter must be reached when the percentage is still very small, of a different order of magnitude even from Rohland's 15 per cent.

G. C. J.

Preliminary Report of the Committee on Coal Analysis of the American Society for Testing Materials and the American Chemical Society. W. A. Noyes and Others. (*J. Ind. and Eng. Chem.*, 1913, **5**, 517-528.)—The Committee appointed a number of sub-committees, each of which now presents an interim report drawn up by its chairman. These interim reports are not endorsed by the committee as a whole, and in some cases do not represent the unanimous opinion of the sub-committee, but only the view of its chairman. They are published mainly to elicit criticism or confirmation of the views now expressed, before bringing the experimental work to a close, and endeavouring to agree on a final report. No novel suggestion is made as regards sampling, but it is proposed to lay down rules as to the size to which coal is to be reduced before each reduction of bulk by quartering. These rules find sanction in the results obtained by Bailey (ANALYST, 1909, **34**, 237) in his mathematical treatment of the subject. Alternative methods are given for determining moisture. The first consists in drying for one hour at 104-110° C. in a current of air dried by means of sulphuric acid. For lignites and coals approaching lignite in character, air must not be used, and the use of dry carbon dioxide is suggested, though the report goes on to say that a current of dry nitrogen is to be preferred in all cases where the highest accuracy is desired. Recognising that these recommendations imply the presence of special apparatus not available everywhere, the committee propose to make permissive drying to constant weight in vacuo at room temperature, but point out that the vacuum must be high—not above 3 mm. of mercury. For volatile matter the seven-minute heating is retained, with a preliminary five-minute "smoking off" period for lignites (*cf.* Cox, ANALYST, 1907, **32**, 298). The manner of heating is changed however, the alternatives offered being—(*a*) heating in a 10 c.c. crucible supported on a tripod in a muffle at 950° C., and (*b*) heating in a 20 c.c. crucible set 1 cm. above the top of a No. 4 Méker burner, having an outside diameter at the top of approximately 25 mm., and giving a flame not less than 15 cm. high, the temperature (900-950° C.) being controlled by a thermocouple or by the m.p. of potassium chromate (940° C.). Ash is to be determined by ignition in a muffle at 700-750° C., or over a small Bunsen flame, but coals containing more than 0·5 per cent. of calcium carbonate are to be treated differently. The ash of such coals is to be sulphated, the amount of carbonate in the coal is to be determined in terms of carbon, and three times this weight of carbon is to be deducted from the weight of sulphated ash. Sulphur may be determined by the Eschka method, by Atkinson's method (*J. Soc. Chem. Ind.*, 1886, **5**, 154), or by the peroxide fusion method, which is most conveniently carried out in the bomb of a Parr calorimeter. It is reported that sulphur is always underestimated (by 3 per cent. or more) when determined in the washings of an oxygen bomb calorimeter. For the determination of calorific power two specifications are given, one of which suffices when an error of 1 per cent. is allowable, the other being applicable to exact work where triplicate determinations may be required to fall within a range of 0·3 per cent. For exact work, the "oxygen" used must contain not less than 95 per cent. of that gas, and the bomb must contain at least 5 grms. oxygen per grm. of coal. A simplified method of computation is recommended. It reduces the calculations to negligible proportions, but the possible error which may result from the substitution of this simplified method of computation for the more closely approximate formulæ of Pfaundler and others is not stated. It must be small, as the committee consider that the results of two analysts should never differ by more than 0·4 per cent.

G. C. J.

Quantitative Separation and Determination of Subsidiary Dyes in the Permitted Food Colours. W. E. Matthewson. (*Circular No.* 113, *Bureau of Chem. U.S. Dept. of Agric.*, through *Chem. News*, 1913, **107**, 218-219.)—For the determination of fast red E in amaranth a solution containing about 0·2 grm. of the dye is treated with 5N hydrochloric acid and diluted to 50 c.c. The solution is shaken out with three separate portions of 50 c.c. of amyl alcohol in three different funnels, and the amyl alcohol portions are washed in the funnels with three portions of

50 cc. of $\frac{N}{4}$ hydrochloric acid, these being passed through the series in the same order as was the original solution. The alcohol is next diluted with light petroleum and the fast red removed with very dilute caustic soda solution, and determined by the method of Knecht and Hibbert, using titanium trichloride (ANALYST, 1911, **36**, 80). Approximate figures may be obtained by diluting 2·5 c.c. of the 1 per cent. solution of the dye with 30 c.c. of $\frac{N}{50}$ hydrochloric acid, and shaking with 15 c.c. of amyl alcohol, which is then compared colorimetrically with pure standards. For the approximate determination of new coccin and similar dyes in amaranth 50 c.c. of the 0·02 per cent. solution of the colour is treated with 10 c.c. of $\frac{N}{10}$ benzidene solution (9·2 grms. per litre in $\frac{N}{2}$ hydrochloric acid), well mixed, and allowed to stand exactly two minutes, filtered, and 50 c.c. of the filtrate titrated against titanium trichloride. Blank determinations with known amounts of pure amaranth and new coccin are carried out at the same time. As fast red E and similar colours interfere if this method has been used for their separation, the extracted solution and washings containing the amaranth are combined and taken to dryness in the water-bath to remove the acid and dissolved amyl alcohol. This evaporation does not affect the amaranth. The determination of monosulphonated dyes in ponceau 3R if carried out by means of amyl alcohol and dilute hydrochloric acid gives high results for crocein orange if this dye be present. An accurate separation can be made by first removing the bulk of the ponceau with dilute acid (about $\frac{N}{16}$), and completing the washing with 5 per cent. sodium chloride solution. The procedure is similar to that adopted for fast red E. The monosulphonated dye present in commercial ponceau 3R is probably mainly sodium trimethylbenzene-azo-naphthol-sulphonate derived from diazotised pseudo-cumidin and S-salt. Instead of this crocein orange was used, and figures are given showing that the separation is very accurate.

In the determination of orange II in orange I advantage is taken of the greater solubility of the latter in dilute sodium carbonate solution, amyl alcohol and a 5 per cent. solution of this alkali being employed as described for fast red E. The equilibrium between the concentrations of the orange I in the two layers is not very quickly established, and the mixtures must be well shaken and given some time to separate, or they may be very quickly separated with a centrifuge.

Determined by these methods in the dyes intended for sale as food colours, amaranth contains from 1 to 5 per cent. lower sulphonated colours, and somewhat more new coccin and similar dyes. Ponceau 3R contains from 1 to 5 per cent. lower sulphonated dyes, and orange I the same amount as orange II.

H. F. E. H.

Identification of Small Amounts of Dyestuffs by Oxidation with Bromine. W. E. Matthewson. (*Chem. News*, 1913, **107**, 265.)—A small quantity of the dye solution is treated with bromine water drop by drop, until about twice as much has been added as is required to destroy the colour. Hydrazine sulphate is then added to take up the excess of bromine, and finally an excess of sodium carbonate. A second test portion is treated similarly, except that a few drops of a 10 per cent. solution of α-naphthol in 50 per cent. alcohol are added before the sodium carbonate. In general, it may be said that if, in testing unknown solutions, the dye is bleached by bromine and restored by hydrazine, or if the coloration with α-naphthol and sodium carbonate is different from that with sodium carbonate alone, a coal-tar dye is present. A concentration of the dye of 1 in 10,000 is suitable for the test, except with some basic dyes which give precipitates which tend to obscure colour reactions. In such cases a greater dilution is preferable. The solution to be tested should be neutral or faintly acid, preferably with hydrochloric acid, which may be present in considerable concentration without interfering. The reactions of fifteen coal-tar colours and three naturally occurring colouring matters are given in tabular form in the paper. G. C. J.

Method of Distinguishing Natural from Artificial Colouring Matters by means of their Electrical Conductivity. G. W. Chlopin and P. J. Wassiljewa. (*Zeitsch. Unter. Nahr. Genussm.*, 1913, **25**, 596-598.)—The authors outline a method which depends on the fact that vegetable and animal colouring matters exhibit a much larger electrical resistance than do artificial colouring matters. The difference in resistance is generally greater in alcoholic than in aqueous solution. For instance, in alcoholic solution, taking the resistance of picric acid as unity, saffron has a resistance of 6, quercitin of 27, etc.; in aqueous solution, cochineal exhibits a resistance 18 times greater than picric acid, and 4 times greater than auramine or chrysoidine. The method will possibly be of use in the analysis of mixtures of colours.

W. P. S.

International Commission for Unification of the Testing Methods of the Stability of Explosives, Final Report (July 25, 1912). (*Eighth Inter. Cong. App. Chem.*, 1912, vol. 25, 305.)—The diverse requirements and natures of explosives in different countries have made it impossible to lay down any fixed rules, but the Commission recommends that for the transport of industrial explosives the packing should be made uniform, and that the following three tests, the first of which would eliminate at once explosives unfit for transport, should be applied: (1) Preliminary test: Two samples, not dried, of 10 grms. each are warmed in loosely covered glass capsules 35 mm. in diameter and 50 mm. high, at 75° C. for forty-eight hours. There must be no decomposition, or alteration in appearance or smell, or evolution of red fumes from nitro-explosives. (2) Shock test: The explosive, powdered and dried, is compared with an equal weight of similarly prepared picric acid; 5 to 10 cgrms. are placed in a $\frac{1}{2}$-inch layer on a steel anvil, and covered with a steel cylinder half an inch in diameter and the same in height. The average fall-height for explosion is determined from ten consecutive tests, and must not exceed that for picric acid. (2*b*) Friction test: The sample must be at least as insensitive as picric acid when rubbed in an unglazed china mortar warmed to 20-30° C. (3) Fire-resistance test: (*a*) Explosives which when powdered take fire in a glass tube from a fuse burning at 1 cm. per second are regarded as easily inflammable; (*b*) explosives passing (*a*) are powdered and poured in quantities increasing from 0·5 to 5 grms. into a red-hot hemispherical iron capsule 12 cms. in diameter and 1 mm. thick; (*c*) after preliminary trials 100 grms. of explosive are tested on an asbestos sheet with an iron bar heated to about 900° C.; it must burn slowly, without explosion, and should go out on removing the bar. Explosives will fall into various classes according to their behaviour under these tests. O. E. M.

Methods for the Determination of the Effective Strength of High Explosives. A. M. Comey and F. B. Holmes. (*Eighth Inter. Cong. App. Chem.*, 1912, vol. 25, 217.)—The lead block compression test gives results which depend upon the rate of detonation and not on the power. More accurate determinations of the rate of detonation are effected by means of the Dautriche and Bichel tests. For determining the power, the ballistic mortar gave the best results. The Druckmesser measures the pressure directly, giving valuable results, while the Trauzl block gives figures only comparable for the same nature of explosive, and affected by the velocity of detonation. The ballistic mortar has been standardised at the Eastern Laboratories, and is there used almost exclusively for power-determinations. O. E. M.

Detection and Estimation of Formic Acid. H. Fincke. (*Biochem. Zeitsch.*, 1913, **50**, 253-288.)—The material to be examined is treated with sufficient tartaric acid to liberate all volatile acids completely, the mixture heated on water bath, and steam passed through, and the issuing vapour passed through a flask containing a suspension of calcium carbonate in boiling water and then condensed. The calcium carbonate suspension is filtered and washed, and the formic acid in the filtrate estimated by the mercuric chloride method. Sulphurous, sorbic, glyoxylic, and lævulinic acids are disturbing factors, as also are several acids of the aromatic series. If sulphurous acid is present, the calcium carbonate filtrate is treated with 1 c.c. $\frac{N}{1}$ sodium hydroxide and 5 c.c. hydrogen peroxide, allowed to stand four hours, and the excess of peroxide decomposed by adding an excess of freshly precipitated mercuric oxide. After standing for half an hour, the liquid is filtered, and the filtrate neutralised with hydrochloric acid. Sorbic acid occurs only in unripe service berries. Glyoxylic acid, if present, colours the suspended calcium carbonate yellowish-brown; it is rendered harmless by gentle boiling with zinc and sulphuric acid for some hours. Lævulinic acid forms a mercury salt readily soluble in hydrochloric acid; it can, therefore, be removed by treating the calomel precipitate with hydrochloric acid. Cinnamic acid does not interfere if the liquid be filtered hot. Salicylic acid gives no precipitate if sodium chloride is added; fumaric acid requires addition of sodium chloride and subsequent treatment with hydrochloric acid, as in the case of lævulinic acid. The formate solution thus prepared is made faintly acid with hydrochloric acid, sodium acetate, sodium chloride, and mercuric chloride added, and the mixture heated for two hours on a water-bath in a flask to which a reflux tube is attached. The mercurous chloride is dried at 100° C. and weighed. If formic acid is present both as free acid and as esters, as in brandies, the slightly acid liquid is treated with 1 to 2 grms. sodium acetate and distilled. The residue is then acidified with tartaric acid, 2 grms. for each grm., sodium acetate being added, and the free acid determined as above. The distillate which contains the volatile esters is saponified with alkali, and the formic acid produced is estimated. The detection and estimation of minute amounts of formic acid is rendered difficult owing to the production of formic acid by the decomposition of sugars on boiling. In order to distinguish between ready-formed formic acid and that produced by decomposition of carbohydrates, successive fractions of the steam distillate are examined separately; if the formic acid is ready formed, the concentration of successive fractions will diminish. If it is being formed during the distillation, the successive fractions will contain the same or increasing amounts of formic acid, provided that the proportion of sugar to decomposition product is large. The paper contains a useful critical review of known methods. E. W.

Estimation of Lactic Acid. A. Bellet. (*Bull. Soc. Chim.*, 1913, **13**, 565-572.) —The process consists essentially in extracting the lactic acid with ether and then converting it into acetaldehyde by the action of potassium permanganate, the acetaldehyde being collected in alkaline silver nitrate solution. The solution or extract containing lactic acid is treated with acid mercuric nitrate solution and heated to precipitate proteins. After filtration, the filtrate is neutralised with sodium hydroxide, evaporated to a syrup, and mixed with anhydrous sodium sulphate and sand, so as to form a dry powder; 2 c.c. of dilute sulphuric acid are added to liberate the lactic acid. The powder is then extracted with ether for three hours in a Soxhlet apparatus, which has a tap on its stem in order to allow the ether to flow back continuously into the extraction flask. The ethereal solution is evaporated, the residue is dissolved in water, transferred to a flask, diluted to 200 c.c., and acidified with sulphuric acid. The flask is connected with a condenser, and a set of receiving vessels are provided containing a known quantity of alkaline silver nitrate solution (silver nitrate, 15 grms.; ammonia, 150 grms.; sodium hydroxide solution, 100 c.c.; and water to 500 c.c.). The solution in the flask is boiled, and a 1·5 per cent. potassium permanganate solution is added drop by drop from a tapped funnel at such a rate that each drop is decolourised before the next is admitted. When the permanganate added is no longer reduced, the contents of the receivers are mixed, filtered through asbestos and glass-wool, and the excess of silver is titrated in the filtrate. One molecule of silver is equivalent to one molecule of lactic acid or one molecule of acetaldehyde according to the equation—

$$Ag_2O + CH_3.CHO = Ag_2 + CH_3.COOH.$$

Succinic, oxalic, and β-hydroxybutyric acids, if present in the material under examination, are extracted together with the lactic acid, but they do not interfere with the estimation of the latter, as succinic acid is not attacked by the permanganate, oxalic acid yields carbon dioxide, and β-hydroxybutyric acid gives acetone.

W. P. S.

New Method for the Identification of Methyl Alcohol. P. N. Raikow. (*Eighth Inter. Cong. App. Chem.*, 1912, vol. 25, 417.)—The methyl alcohol is converted into nitromethane, which yields with nitroprusside a blue, and on standing a green, and finally a yellow, coloration. The sample (200 c.c.) is acidified with phosphoric acid, and 10 c.c. distilled. The distillate is treated with 4 grms. of red phosphorus and 25 grms. of iodine under a reflux condenser. After twenty minutes the mixture is fractionated, and the first 5 c.c. of distillate slowly distilled with 2 to 3 grms. silver nitrite. The distillate is collected in portions of 3 to 4 drops, and to each of these strong ammonia and a small quantity of concentrated sodium nitroprusside solution added. The presence of acetone, which gives a colour similar to Hofmann's violet, does not vitiate the test. O. E. M.

Colour Reaction of Proteins. L. Lewin. (*Ber.*, 1913, **46**, 1796-1798.)—Many proteins, even in very dilute solution, give a violet coloration when treated with a 0·1 per cent. solution of triformoxime (Kahlbaum's trioximinomethylene) in unpurified sulphuric acid, or in any concentrated sulphuric acid to which a trace of selenious acid has been added. The reaction is given distinctly by egg albumin and Witte's peptone in a dilution of 0·05 per cent., and a solution of half this strength gives a coloration which is easily seen against a white background. Serum albumin, casein, pepsin, and nucleoproteins also give the reaction, but gelatin does not, in any concentration. Indol gives a similar colour ($\lambda = 536$ $\mu\mu$), and some alkaloids—for example, codein—give a blue colour with the reagent. Paraformaldehyde may be substituted for the reagent, but is less satisfactory, the intensity of coloration produced being somewhat less, and the use of a large excess of paraformaldehyde completely inhibits the production of the violet coloration. G. C. J.

Polarimetric Method for the Determination of Starch in Paper. C. E. G. Porst and H. A. Crown. (*Eighth Inter. Cong. App. Chem.*, 1912, vol. 26, 13-16.)—The specific rotatory power of maize starch when determined in 0·5 per cent. solution was found to be $[\alpha]_D^{20} = 187·9°$. A modification of Lintner's polarimetric method was employed (ANALYST, 1909, **34**, 332; *ibid*, 1912, **37**, 498), using 5 grms. of paper moistened with 20 c.c. of water in an ice-cold mortar. Forty c.c. of ice-cold concentrated hydrochloric acid are added, and the whole well macerated. After standing at 20° C. for thirty minutes the mixture is washed into a 200 c.c. flask with acid of 1·125 sp. gr., and 10 c.c. of 4 per cent. phosphotungstic acid are added. After making up to 200 c.c. the contents of the flask are kept at 20° C. for half an hour, filtered, and the reading taken in a 400 mm. tube fifteen minutes after the filtration has begun; 1° V. is equivalent to 1·845 per cent. starch. H. F. E. H.

The Determination of Tartaric Acid. P. B. Dunbar. (*Eighth Inter. Cong. App. Chem.*, 1912, vol. 26, 361-373.)—When a solution of tartaric acid or tartrates containing between 0·2 and 3·0 grms. of total tartaric acid per 100 c.c. is treated under definite conditions with uranyl acetate, its optical rotation is much increased (ANALYST, 1911, **36**, 498), and is proportional to the amount of tartaric acid present in the solution. A measured volume of the tartaric acid solution is made slightly alkaline to litmus with soda, 5 grms. of sodium acetate are dissolved in it, and the solution reacidified in slight excess with strong citric acid, and the whole diluted to 100 c.c. Powdered uranyl acetate is added to this solution, the mixture being mechanically agitated for three hours, enough of the salt being used to leave some undissolved at the end of this period. Under these conditions each grm. of tartaric acid per 100 c.c. produces a rotation of +19·6° V. in a 200 mm. tube. A standard Lippich type triple field saccharimeter is employed with white light. The reading so obtained, if multiplied by 0·051, gives the percentage of tartaric acid, unless malic acid or other optically active substance is present, in which case a portion of the solution must be freed from tartaric acid by precipitation with dry powdered normal lead acetate and polarised separately. The algebraic difference between this reading and that obtained in the uranium-treated solution is the reading due to the uranyl tartaric complex. Very dark-coloured solutions may often be cleared by vigorous shaking with a few drops of bromine, followed by filtration just before reading.

H. F. E. H.

Determination of Malic and Tartaric Acids in the Same Solution.

P. B. Dunbar. (*Eighth Inter. Cong. App. Chem.*, 1912, vol. 26, 375-386.)—The author and R. F. Bacon have described a method (ANALYST, 1912, **37**, 29) for the estimation of malic acid based on the increase in rotation produced by the addition of uranyl acetate, and as an extension of this work (see preceding abstract) suggested a method for the estimation of malic and tartaric acids when present together depending on the facts that the optical rotation of both acids is increased independently of the other when treated with uranyl acetate under definite conditions, and that both acids may be oxidised quantitatively to oxalic acid by heating with alkaline permanganate. The presence of any other substances which form oxalic acid on oxidation incapable of being removed before treatment with permanganate interferes with the method. Under the conditions described in the previous abstract, it was shown that 1 grm. of tartaric acid per 100 c.c. produced a rotation of $+19{\cdot}61°$ V.; and as described by the author and Bacon (*loc. cit.*), 1 grm. of malic acid per 100 c.c. will rotate $-27{\cdot}77°$ V. One grm. of potassium permanganate is equivalent to 0·475 grm. of tartaric acid, or 0·353 grm. of malic acid, while 1 grm. of oxalic acid is equivalent to 0·526 grm. of malic acid, or 0·595 grm. of tartaric acid. One grm. of malic acid equals 1·90 grm. of oxalic acid, or 2·8297 grms. of potassium permanganate, while 1 grm. of tartaric acid equals 2·1062 grms. of permanganate, or 1·68 grms. of oxalic acid. If x = grms. of malic acid in 100 c.c., y = grms. of tartaric acid in 100 c.c., a = polarisation in degree V. after treatment with uranyl acetate and b = grms. of permanganate required to oxidise the acids in 100 c.c.; then—

$$x = -0{\cdot}0185a + 0{\cdot}1720b,$$
$$y = 0{\cdot}0248a + 0{\cdot}2436b;$$

while if the amount of oxalic actually found is used instead of the permanganate required to oxidise this acid, the equations become—

$$x = 0{\cdot}020a + 0{\cdot}233c,$$
$$y = 0{\cdot}023a + 0{\cdot}331c,$$

when c is equal to the grms. of oxalic acid formed by oxidation of the acids in 100 c.c.

H. F. E. H.

INORGANIC ANALYSIS.

Application of Folin's Method for the Determination of Ammonia to Fertilisers.

O. Folin and A. W. Bosworth. (*J. Ind. and Eng. Chem.*, 1913, **5**, 485.)—Two grms. of fertiliser are placed in a 100 c.c. graduated flask, about 50 c.c. of water added, and then 25 c.c. of approximately $\frac{N}{1}$ hydrochloric acid. The volume is made up to 100 c.c. with water, the contents of the flask are shaken, and after a few minutes' standing are shaken again. The flask is next allowed to stand till the heavier undissolved particles have settled; then 5 c.c. of the supernatant liquid are withdrawn and transferred to the tube of the Folin apparatus (*J. Biol. Chem.*, **11**, 493). Two c.c. of a saturated solution of potassium oxalate, a few drops of kerosene, and then 2 c.c. of a saturated solution of potassium carbonate are then added. The apparatus is at once closed, and air is passed through for ten to twenty minutes. The ammonia is collected in 20 c.c. of $\frac{N}{10}$ hydrochloric acid. If the air current is produced by a blast, the ammonia is collected in a flask, and 25 c.c. of water are added to the 20 c.c. of acid to prevent loss of ammonia. The ammonia is titrated against $\frac{N}{10}$ soda, using alizarin red as indicator. The results agree well with the values obtained when the sample is distilled with magnesium oxide in the ordinary way. H. F. E. H.

Estimation of Sulphate in Ammonium Sulphate Solution, with Special Reference to the Testing of Illuminating Gas. R. S. McBride and E. R. Weaver. (*J. Ind. and Eng. Chem.*, 1913, **5**, 469-474.)—For the determination of sulphate, in cases where an approximate estimation is all that is required, the following turbidimetric method is recommended. The solution is neutralised with hydrochloric acid, of which an excess of about 1 c.c. is then added. Paranitrophenol is the most convenient indicator to use, as the acid solution is then colourless. The solution is made up to 270 c.c., 360 c.c., or other multiple of 90 c.c., and 90 c.c. is then mixed at 25 to 30° C. with 10 c.c. of 10 per cent. barium chloride solution. After stirring a minute, the mixture is poured slowly into a graduated glass cylinder, with polished bottom, and contained within an outer copper cylinder, until the filament of a 16 c.p. carbon filament lamp, fixed below the apparatus, can no longer be seen. The height of the liquid in the cylinder is then measured, and its content of sulphate sulphur determined by reference to a curve, constructed from the results of experiments with known quantities of sulphuric acid under the same conditions, among which that of temperature is the most important. Between the concentrations of 2 and 11 mgrms. sulphate sulphur per 100 c.c., corresponding to a measured height of liquid of from 110 to 25 mm., the extreme error of a single determination is 7 per cent. The use of oxalic acid and other additions to the barium chloride, as has been recommended by some authors, is not advisable. Such additions give a more opaque mixture, but duplicate determinations are more discordant.

A volumetric method, capable of giving results never so much as 1 per cent. in error, is the following: The solution is diluted or concentrated to 300 c.c., neutralised, and then acidified with 10 c.c. of dilute (1 : 1) hydrochloric acid, and heated to boiling. Exactly 15 c.c. of barium chloride solution (50 grms. crystals per litre) is added, the mixture boiled for five minutes, and then exactly 15 c.c. of a chromate solution, rather more than equivalent to the barium solution (*e.g.*, 30·5 grms. potassium bichromate per litre), is added, together with a few drops of ferric chloride solution. Ammonia is added until a permanent precipitate appears, when a further 5 c.c. of 10 per cent. ammonia is added, the mixture is boiled, filtered and washed with hot water, the ferric hydroxide facilitating these processes. To the cooled filtrate, acidified with hydrochloric acid, 2 grms. potassium iodide are added, and the liberated iodine is titrated with thiosulphate. A similar blank experiment is made, starting with 300 c.c. distilled water. The consumption of thiosulphate in the blank test is subtracted from that in the determination proper, and the remainder calculated to sulphate sulphur. $S = 3Na_2S_2O_3$. The method does not differ in principle from methods previously described, but its greater accuracy is due to the use of the blank test. In methods depending on the use of a hydrochloric acid solution of barium chromate, the reducing action of the acid on the chromate has introduced errors. In others depending on the use of exactly equivalent solutions of barium chloride and potassium chromate, any inexactness in strength has introduced error, whilst methods depending on the use of solid barium chromate require a salt absolutely free from alkali chromate, and this is not readily obtainable in commerce. All methods of this type are affected by the solubility of barium chromate. The use of a chromate solution slightly stronger than the barium solution, together with a blank test, eliminate all these sources of error, and the small error that remains (1 per cent.) is mainly due to the smallness of the quantity (6 to 30 mgrms.) to be estimated. G. C. J.

Separation of Cadmium from Zinc. W. D. Treadwell and K. S. Guiterman. (*Zeitsch. anal. Chem.*, 1913, **52**, 459-470.)—The separation of cadmium from zinc, by means of hydrogen sulphide, is best effected in sulphuric acid solution. In hydrochloric acid solution much more zinc is co-precipitated with the cadmium. Cadmium sulphide is quite insoluble in 6 N sulphuric acid at 18° C., whereas at 70° C. it is soluble in 2 N acid. The best results are obtained by making the solution 4 to 5 normal with sulphuric acid, heating to 80° or 90° C., and then allowing the solution to attain the temperature of the room whilst passing a current of hydrogen sulphide through it. Precipitation of cadmium is then quantitative, and the quantity of zinc co-precipitated is negligible if zinc is present in amount only equal to the cadmium. In presence of relatively large quantities of zinc, separation cannot be effected by a single precipitation. For example, 0·1 grm. of cadmium, when precipitated as sulphide in presence of 2 grms. of zinc, will take down with it 6 mgrms. of zinc. When, therefore, cadmium has to be estimated in presence of more than its own weight of zinc, the precipitated sulphides are re-dissolved in hot dilute hydrochloric acid, the solution is evaporated to dryness, the chlorides are decomposed by heating with sulphuric acid, and the cadmium is finally re-precipitated as sulphide.

Small quantities of cadmium can be separated from quantities of zinc not exceeding 1 grm. by electrolysis in oxalate solution. To the neutral solution of the chlorides or sulphates, 8 grms. potassium oxalate, 2 grms. ammonium oxalate, and 0·4 grm. oxalic acid are added, and the solution is diluted to about 120 c.c. It is then heated to 70° to 80° C., and electrolysed by a current of 0·03 ampère, corresponding to a P.D. between the terminals of about 1·5 volts. Some four or five hours are required to deposit 0·1 grm. cadmium, and the electrolysis is most conveniently carried out in a beaker nearly filled with the electrolyte, a round-bottomed flask, through which water is passed, can be set on such a beaker so as to touch the liquid, and serves the double purpose of preventing evaporation and inducing circulation of the liquid.

Quantities of cadmium between 0·04 and 0·2 grm. can be separated from fifty times their weight of zinc by electrolysis in cold sulphuric acid solution. The neutral solution of the sulphates is most conveniently acidified by addition of 5 grms. of sodium bisulphate. A rotating cathode is used, and a constant cathode potential of 2·5 volts corresponding to a current of from 0·3 to 0·05 ampère. More than 0·2 grm. can be deposited in fifty minutes. The method is less troublesome than the oxalate method, but for very small quantities of cadmium it is less accurate.

G. C. J.

Rapid Method for Estimating Carbon in Iron and Steel. E. Szász. (*Zeitsch. angew. Chem.*, 1913, **26**, 281-286.)—The method and apparatus described have been designed with a view to making an accurate determination of carbon in any iron, steel, or steel-making alloy in the shortest possible time; and, since they accomplish this purpose, the author does not hesitate to recommend an apparatus which is both costly and complicated. Once set up, the apparatus is extremely easy to manipulate, and, apart from the fact that platinum apparatus has a high scrap value, it is suggested that the capital locked up in this apparatus will be highly remunerative in many cases—for example, in controlling an electric furnace utilising mixed scrap. A carbon determination can be completed in less than ten minutes. The principle of the method is direct, dry combustion, with measurement of the carbon dioxide produced. A necessary condition of such a method is the use of as small an excess of oxygen as possible, since otherwise a gas burette of inconveniently large dimensions would be required, and another essential is to have the dimensions of the combustion apparatus as small as possible. The combustion chamber is constructed of platinum, with water-jacketed platinum tubes for the introduction of oxygen and egress of gases, and a water-jacketed stopper. It is only just sufficiently large to accommodate the boat and the surrounding platinum cylinder which safeguards the combustion chamber walls from damage by spirting. The use of an unnecessary excess of oxygen is avoided by taking advantage of the well-known fact that, when a certain temperature is reached in the combustion of iron in oxygen, there is a very rapid absorption of oxygen, so rapid as to require prompt manipulation of the oxygen supply, or special appliances (*cf.* Blount and Levy, ANALYST, 1909, **34**, 93) designed to adjust the supply automatically. The author charges his apparatus with oxygen after inserting the boat, disconnects the train of apparatus leading to the measuring burette by turning a cock, but leaves the combustion apparatus connected to a supply of oxygen through an apparatus which guards against any backward movement of gas during the first stage of heating. The combustion apparatus is then heated by means of a Méker burner supplied with producer gas and air under pressure. In one and a half minutes or less the period of rapid absorption of oxygen is reached (over 1,000° C.), and at the end of this one and a half minutes the cock connecting with the measuring burette is opened, and its evelling cylinder is lowered at such a rate that the burette (200 c.c., with 150 c.c. bulb and 50 c.c. divided stem) is nearly filled with gas at the end of a further two or three minutes. The gas in the water-jacketed burette is then measured, transferred to a potash pipette, returned and remeasured. The temperature of the combustion apparatus continues to rise after the expiration of the preliminary period of one and a half minutes, and reaches 1,150°-1,200° C. after two minutes or a little longer. The supply of oxygen is freed from carbon monoxide, said to be often present in commercial oxygen, by passing it through a heated quartz tube containing thin platinum wires, before conducting it to the usual potash vessel. The gases resulting from the combustion of the iron are also passed through a heated quartz tube containing copper oxide to oxidise any carbon monoxide, platinum failing in this position in absence of excess of oxygen. The gases are deprived of sulphur dioxide by passing over glass beads moistened with a few drops of chromic acid solution, and contained in a water-jacketed U-tube which serves to cool the gases on their way to the burette. Some sixty test numbers given do not differ from the results obtained on the same material by proved gravimetric methods by more than the difference between duplicate gravimetric results. Some special steels and steel-making alloys require slightly special treatment, but, of the fifteen types of material experimented with, none has failed to yield the whole of its carbon as carbon dioxide. On the use of diluents to keep the material open, oxidising agents, and materials to hold back volatile metallic oxides in the boat, there are some notes which apply to the direct dry combustion of special steels and steel-making alloys generally, and not merely to the present modification of that process. G. C. J.

Estimation of Carbon Dioxide. L. W. Winkler. (*Zeitsch. anal. Chem.*, 1913, **52**, 421-440.)—Apart from a modification of Pettenkofer's apparatus for the determination of carbon dioxide in air, there is nothing new in this paper, but within its comparatively narrow limits will be found descriptions of methods and illustrations of apparatus by means of which carbon dioxide can be determined in almost any material with satisfactory accuracy. Half the paper is devoted to a detailed description of the author's method and apparatus for the estimation of total carbon dioxide in natural waters (*cf. Zeitsch. anal. Chem.*, 1903, **42**, 735), and to showing that the criticism to which that method has been subjected is not well founded. His well-known method (*Zeitsch. anal. Chem.*, 1901, **40**, 523) for the estimation of carbon dioxide in mineral waters is redescribed in sufficient detail to enable anyone to use it, with notes on a simple method of taking and preserving samples of such waters. An apparatus for the determination of carbon dioxide in solid carbonates (*cf.* Ekkert, *Chem. Zeit.*, 1905, **29**, 1316) is also described and illustrated, and shown to afford a means of making this determination with great accuracy and at the same time with the minimum expenditure of time and attention. Finally the following modification of Pettenkofer's apparatus is described. It consists of a hollow stopper for the large bottle employed by Pettenkofer. The stopper has approximately the form of an 8-inch × 1-inch test-tube, constricted about 3 inches from its mouth, which is ground to serve as the stopper. The capacity of the stopper above a mark on the constricted portion is 50 c.c., and before the analysis it is inverted and filled to the mark with lime-water, after which it is pressed into the neck of the inverted bottle. After agitation and long standing, the liquid is allowed to collect again in the hollow stopper as far as possible, the stopper is removed, plugged at the constriction by a rubber stopper mounted on a glass rod, and when the precipitate has settled, leaving the liquid clear, 25 c.c. of the latter is pipetted off and titrated in the usual manner.

G. C. J.

Gravimetric Estimation of Carbon Dioxide. A. Dejeanne. (*Bull. Soc. Chim.*, 1913, **13**, 556-560.)—The carbon dioxide liberated in the usual way from a carbonate, etc., is absorbed in a definite quantity (an excess) of barium hydroxide solution; magnesium chloride solution is then added, the mixture is diluted to a definite volume, filtered, and the excess of barium in the filtrate is estimated by sulphating an aliquot portion of the latter. The addition of the magnesium chloride is for the purpose of converting the excess of barium hydroxide into barium chloride, a corresponding quantity of magnesium hydroxide being precipitated and filtered off with the barium carbonate; precautions to exclude atmospheric carbon dioxide during the filtration are unnecessary.

W. P. S.

Iodimetric Estimation of Free and Combined Chromic Oxide. M. Gröger. (*Zeitsch. anorg. Chem.*, 1913, **81**, 233-242.)—Chromic oxide and most of its compounds, except chrome ironstone, are decomposed by nitric acid and potassium chlorate, with complete solution of the chromium as chromate. This method of decomposition is therefore preferred to nitre fusions, which involve the somewhat rapid destruction of platinum crucibles. It is shown that the accuracy of the results often obtained by precipitating the chromic acid as barium chromate is due to compensation of errors, which in many cases do not exactly balance each other, but result in a net error of as much as ± 2 per cent. An iodimetric determination of the chromate, on the other hand, need not be in error by as much as corresponds to 0·05 c.c. of $\frac{N}{10}$ thiosulphate. The substance (0·3 grm.) is heated on the water-bath with 10 c.c. of concentrated nitric acid contained in a 300 c.c. flask closed with a small funnel, and about 1 grm. of potassium chlorate is added in small portions at a time, the additions being spread over about one hour. The funnel is removed, heating continued for an hour to expel chlorine, the solution cooled and diluted to 250 c.c., and 50 c.c. treated with excess of potassium iodide and titrated with thiosulphate. Heating for an hour suffices to expel chlorine, as portions of the solution evaporated to dryness before treatment with iodide yield substantially identical results. A warning is given that 10 c.c. of nitric acid sold as "chemically pure" may contain as much ferric salt as corresponds to 0·05 c.c. $\frac{N}{10}$ thiosulphate. The method gives results of the accuracy stated in presence of large quantities of calcium, barium, strontium, magnesium, zinc, cadmium, aluminium, nickel, and cobalt, and artificial chromites containing magnesium, zinc, cadmium, and nickel, gave quantitative results, but required about 1·5 grms. of chlorate and about two hours for complete decomposition. Chrome ironstone is not completely decomposed by the acid mixture. Iron existing in any form other than chromite can be eliminated, like copper, by precipitation with hot caustic alkali and filtration, the whole of the chromic acid passing into the filtrate, in which its estimation iodi-

metrically presents no difficulty. In presence of manganese, the method fails owing to the formation of an insoluble compound of manganese dioxide and chromic oxide.
G. C. J.

Quantitative Separation of Iron and Chromium. F. Bourion and A. Deshayes. (*Comptes rend.*, 1913, **156**, 1769-1771.)—The method described by Bourion for the separation of iron and titanium, depending on the volatilisation of the former in a current of hydrogen chloride and sulphur monochloride (ANALYST, 1912, **37**, 323), is not applicable to the separation of iron and chromium. In presence of chromium some iron always remains unvolatilised. Since anhydrous chromic chloride is insoluble in water, it was thought that a separation might be effected by converting the mixed oxides into chlorides by the action of chlorine and sulphur dichloride, and subsequent treatment with water. Unfortunately, chromic chloride is not quite insoluble in solutions of ferric chloride; but in mixtures containing not more than 30 per cent. of chromium the latter element can be determined with an error not exceeding $-0{\cdot}4$ per cent. by proceeding as follows: The mixed oxides, contained in a boat in a combustion-tube, are heated gradually from 200° to 650° C., whilst a slow stream (5 to 15 bubbles per minute) of chlorine, saturated at 30° to 40° C. with the vapour of sulphur dichloride, is led through the tube. No ferric chloride remains in the boat, but some of the chromic chloride is volatilised and condenses on the cooler portions of the tube. The boat is withdrawn and weighed, and the contents of the tube are rinsed out and filtered through a Gooch crucible to recover the volatilised chromic chloride. When the proportion of chromic oxide in the mixture to be analysed exceeds 40 per cent., the error of the above method increases, and 95 per cent. of chromic oxide might be under-estimated by as much as 5 per cent. Any mixture can be analysed with an error not exceeding $-0{\cdot}4$ per cent. by proceeding as follows: The mixed oxides are mixed in the boat with an equal volume of ammonium sulphate before commencing the experiment. The mixture is then heated gradually (three to four hours) to 650° C. in a current of chlorine and sulphur dichloride, and the volatilised portion of the chromic chloride is separated from ferric chloride as already described. The ammonium sulphate fuses before decomposing, and the resultant mass is of such a character that the amount of chromic chloride volatilising is small, and that which does volatilise condenses as a compact mass close to the boat, this compact mass being less easily brought into solution by ferric chloride than the minute crystals which spread over a considerable length of the tube when ammonium sulphate is not used. G. C. J.

Volumetric Estimation of Chromium in Presence of Iron. A. Kurtenacker. (*Zeitsch. anal. Chem.*, 1913, **52**, 401-407.)—The solution, contained in a measuring flask, is treated with excess of bromine-water, then with excess of caustic alkali, heated on the water-bath for half an hour with frequent shaking, cooled, made up to the mark, and filtered. The chromate in an aliquot portion of the filtrate is determined in the manner described later, whilst the precipitate of ferric hydroxide is washed, dissolved in hydrochloric acid, reduced and titrated with permanganate, using Reinhardt's manganese phosphate solution. Before the chromate in the filtrate can be determined iodimetrically, the hypobromite and bromate must be destroyed. The former is readily destroyed by acidifying and heating the solution, when bromine is expelled and more bromate results. In presence of the bromide simultaneously present, the bromate is also decomposed in acid solution, but for the reaction to be quantitative and reasonably quick, the hydrion concentration must not be too low. On the other hand, if it be too high, hydrogen bromide may reduce chromic acid. It is possible to destroy hypobromite and bromate whilst not reducing chromic acid, but the permissible limits of acid concentration are very narrow in the case of hydrochloric acid, and restricted in the case of sulphuric acid. The point of the present communication is the discovery that potassium bisulphate in comparatively low concentration serves to make the destruction of bromate quantitative, whilst even in concentrations ten times as high it does not support the reduction of chromic acid. The chromate solution (100 c.c. or less) is acidified cautiously with sulphuric acid until it acquires a permanent brown colour. About 20 c.c. of 30 per cent. potassium bisulphate is added and the mixture boiled for five minutes. As a rule this suffices to destroy all bromate and expel all bromine, but if any smell of bromine is apparent, a further 10 c.c. of bisulphate solution is added and the boiling repeated. The solution is then cooled, potassium iodide, sulphuric acid, and a considerable amount of water, are added, and the liberated iodine is titrated with thiosulphate. The maximum error recorded corresponds to 0·05 c.c. of $\frac{N}{10}$ thiosulphate, and in some experiments the concentration of the potassium bisulphate in the mixture boiled to destroy bromate was as high as 13 per cent., in others as low as 5 per cent.

G. C. J.

Analysis of Copper-Tin Alloys. W. Gemmell. (*J. Soc. Chem. Ind.*, 1913, **32**, 581-584.)—It is not necessary to remove tin before proceeding to the estimation of copper by electrolysis, provided the tin is kept in solution. Copper-tin alloys are completely soluble in nitrosulphuric acid, and from solutions of suitable concentration the copper can be deposited electrolytically in a pure condition. The alloy (2 grms.) is dissolved in a mixture of 10 c.c. nitric acid (sp. gr. 1·4), 10 c.c. sulphuric acid, and 30 c.c. water; the solution is diluted to about 100 c.c. and electrolysed, using rotating platinum electrodes and a current of about 8 ampères. Deposition of the copper is complete in thirty minutes. The beaker containing the exhausted solution is removed, and immediately replaced by one containing water, without breaking the electrical circuit, and the electrodes are rinsed down with distilled water, immersed in alcohol, dried at 100° C., and weighed. The copper on the cathode contains no trace of tin. If lead be present, some of it is deposited on the anode as peroxide, and some separates as sulphate at the bottom of the vessel. For the determination of tin, the solution which has been electrolysed for copper, together with the water used to wash the electrodes, is poured into a cold 2 per cent. solution of sulphuric acid saturated with hydrogen sulphide. After boiling for a few minutes, the precipitated sulphides are filtered off and washed with 1 per cent. acid saturated with hydrogen sulphide. The tin sulphide is dissolved on the filter by means of freshly-prepared ammonium sulphide, the solution is electrolysed to recover the tin, and any lead sulphide left on the filter-paper can be converted into lead sulphate, and the lead found added to that recovered as peroxide. The test numbers show that 1 or 2 per cent. of lead in a copper-tin alloy can be determined in this manner with an error not exceeding 4 per cent. of the lead present, but the following more exact method is recommended for the determination of small proportions of lead: The alloy (5 to 10 grms.) is dissolved in nitrosulphuric acid, and the solution evaporated until the sulphuric acid fumes. Water is next added until the concentration of acid is about 25 per cent. by volume. Such a solution can be boiled if necessary to insure solution of copper and tin as sulphates, since at this concentration of acid there is no risk of tin being precipitated as oxide. When cool, the solution is diluted until the acid is at a concentration of about 7 per cent. by volume, and after standing, preferably overnight, the lead sulphate is filtered off, dried, ignited, and weighed. Most commercial copper alloys contain nickel, the proportion of which may exceed 0·4 per cent. without any intentional addition of the metal. After precipitation of the tin from the solution which has been electrolysed for copper, nickel and iron are determined as follows: The filtrate from the tin sulphide is boiled to expel hydrogen sulphide, aqua regia is added to oxidise iron, and iron and nickel are precipitated together by addition of caustic soda. The precipitated hydroxides are filtered off, washed, dissolved in hydrochloric acid, excess of ammonium chloride is added, and the iron reprecipitated by addition of ammonia. In the filtrate from the ferric hydroxide, nickel is determined by precipitation by means of dimethylglyoxime. Zinc is determined in the filtrate from the hydroxides of nickel and iron. This filtrate is acidified with sulphuric acid and evaporated until fumes arise, cooled, diluted, made alkaline with caustic soda, and then acidified with acetic acid. The zinc is deposited electrolytically on a platinum electrode which has been previously plated with copper. A current of 3 ampères suffices to remove 0·7 grm. zinc in less than forty minutes. The test numbers are good, the highest error recorded being one of +0·08 per cent. on the alloy, representing an error of 2 per cent. on the constituent estimated (zinc). Maximum error for copper −0·03 per cent. with 80 per cent. present, for tin ±0·04 per cent. with 20 per cent. present, for iron ±0·02 per cent. with 1 per cent. present. The methods are also said to be more rapid than other less accurate ones. G. C. J.

Rapid Estimation of Copper in Burnt Pyrites. H. Koelsch. (*Chem. Zeit.*, 1913, **37**, 753.)—An addition of sodium hypophosphite to the hydrochloric acid causes rapid reduction and solution of the ferric oxide in burnt pyrites. Ten grms. of the finely powdered material are heated with 6 to 7 grms. of solid sodium hypophosphite and 40 c.c. of hydrochloric acid (sp. gr. 1·16), and the solution diluted with 100 c.c. of hot water and treated with 50 c.c. of sodium sulphide solution (40 grms. per litre). The precipitate rapidly agglomerates on shaking, and is filtered after five minutes and washed with hot water. It is then ignited, decomposed with nitric acid, heated with sulphuric acid, washed with 100 c.c. of water into a beaker, and the copper estimated electrolytically. Or a volumetric method may be used, the small proportion of iron not affecting the results of either method. C. A. M.

Use of "Cupferron" in the Separation of Titanium from Aluminium. J. Bellucci and L. Grassi. (*Gazz. Chim. Ital.*, 1913, **43**, 570-576.)—The ammonium salt of nitroso-phenyl-hydroxylamine, $C_6H_5(NO)N.ONH_4$, which Baudisch termed "cupferron," and used as a reagent for the precipitation of copper and iron (ANALYST, 1911, **36**, 520), also precipitates titanium as a bright yellow salt, $[C_6H_5(NO)NO]_4Ti$, which on ignition readily yields a residue of titanium oxide, TiO_2. It is essential that the solution of the titanium salt should be decidedly, but not excessively, acid before the addition of the reagent. The precipitate is collected, washed repeatedly with cold water, and ignited in a tared porcelain crucible. In using the method for the separation of titanium from aluminium, the precipitate must be washed, first by decantation and then on the filter, with very dilute hydrochloric acid in order to remove all traces of aluminium. The method is shown to give good results with mixtures containing 1 part of titanium to 50 of aluminium. In another portion of the solution the titanium and aluminium are precipitated simultaneously as hydroxides, and weighed together as $Al_2O_3 + TiO_2$. The reagent may also be used for the separation of titanium from chromium, nickel, cobalt, manganese, etc. C. A. M.

Reduction of Ferric Salts, and their Titration with Potassium Permanganate. A. Leclère. (*J. Pharm. Chim.*, 1913, **7**, 587-589.)—The method is based upon the formation of the stable ferrous ammonium sulphate in the solution of the ferric salts. The liquid is treated with a fragment of granulated zinc and of platinum wire, and acidified with 1 to 2 per cent. of sulphuric acid. From 2 to 3 grms. of ammonium sulphate are then added, and the flask gently heated for at least an hour upon the water-bath or over a small flame, until the reduction of the iron is complete. The solution of ferrous salt is filtered through glass-wool, and the filtrate and washings titrated with standard permanganate solution in the usual way.

C. A. M.

Volumetric Determination of Gold. V. Lenher. (*J. Amer. Chem. Soc.*, 1913, **35**, 733-736.)—Auric chloride when treated with potassium iodide is quantitatively reduced to aurous chloride, with liberation of iodine, which can be titrated with standard sulphurous acid. The results are exact, as are those obtained by substituting potassium bromide for potassium iodide, with subsequent titration of the liberated bromine. If the auric solution to be titrated be first mixed with an equal bulk of a saturated solution of sodium chloride or with magnesium chloride, the colour of the auric solution is intensified, and the titration with sulphurous acid can be conducted without the use of potassium iodide or bromide. The results are exact, but auric solutions cannot be directly titrated in hydrochloric acid solution free from alkali salts. In such circumstances, the colour of the solution is paler to start with, sulphurous acid has no distinct bleaching effect, and metallic gold is readily separated. In applying the above methods to metal, the latter is dissolved by means of chlorine-water or hydrochloric acid and potassium chlorate, and free chlorine is removed by addition of ammonia until a permanent precipitate forms. The solution is then acidulated with hydrochloric acid, warmed until the precipitate dissolves, and the gold is determined by any of the above methods. No attempt must be made to expel chlorine from the gold solution by evaporation, as some aurous chloride always results when this is done.

The sulphurous acid solution is standardised either against stardard iodine solution, or against an acidified solution of potassium iodide to which a measured volume of standard permanganate has been added. By direct titration of sulphurous acid against permanganate, the author was unable to establish the strength of the former within 1 per cent. G. C. J.

Volumetric Estimation of Manganese by the Volhard-Wolff Method. Chemikerkommission des Vereins deutscher Eisenhüttenleute. (*Stahl u. Eisen*, 1913, **33**, 633-642; through *Chem. Zentralbl.*, 1913, I., 2003).—In blank tests the permanganate consumption is the same whether a small or a large excess of zinc oxide is used. Nitric acid solutions give the highest consumption of permanganate, hydrochloric acid less, and sulphuric acid the least consumption. In presence of sulphates the consumption of permanganate is less than in presence of an equivalent quantity of chloride or nitrate. In sulphuric acid solution the consumption of permanganate is greatest when the excess of zinc oxide is very small. In solutions of chlorides or nitrates the amount of zinc oxide present has no measurable influence on the results. The influence of varying concentration of the manganese solution is also negligible. The consumption of permanganate is highest when the titration is made in boiling or almost boiling solution. If the permanganate is added in several portions, halting between each addition, or if the solution be continuously shaken throughout the titration, the consumption of permanganate is too low. In exact work, the removal of the iron precipitate by filtration is essential. Even when iron is removed, manganese may be over-estimated if cobalt is present in quantity. No other metals, in the proportions in which they are likely to be present, interfere. The permanganate solution must be standardised on a solution of known manganese content under conditions closely approximating those obtaining in actual analytical work. G. C. J.

Gasometric Estimation of Nitric Oxide and Oxygen, depending on the Formation of Nitrous Anhydride. G. Klinger. (*Ber.*, 1913, **46**, 1744-1748.)—In an earlier paper (ANALYST, 1913, 45) it was stated that nitric oxide could be oxidised quantitatively to nitrous anhydride, provided it was confined together with some stick potash over mercury before admitting air. Under these conditions no nitrogen tetroxide is formed, since the nitrous anhydride is removed by the potash as fast as it is formed. It was also stated that inaccurate results were obtained when the process was reversed and the nitric oxide led into excess of air or oxygen confined with potash over mercury. It is now shown that these inaccurate results were due, not to excess of oxygen, but to presence of moisture. To obtain accurate results, the gases should be dry, and the potash should not be unnecessarily exposed to the air. A piece of stick, from one of the sealed bottles in which potash is sold for analytical purposes, serves well, but it should come from a bottle which has been kept sealed, and be quickly transferred to the gas pipette. With these precautions, the method is available, not only for the determination of nitric oxide, but for the determination of oxygen by use of an excess of nitric oxide. The criticism of Koehler and Marqueyrol (ANALYST, 1913, 171), that the method is not available in presence of carbon dioxide, is met by the statement that the gas in which nitric oxide is to be estimated can be freed from carbon dioxide and its content of that gas determined by the use of the same potash pipette that is used in the nitric oxide estimation. The use of this apparatus containing solid potash for absorbing carbon dioxide has the advantage that it does not, like a potash solution, absorb nitrous oxide, should that gas be present in the mixture under analysis. G. C. J.

Estimation of Water-Soluble Phosphates in Ammonium Superphosphates. E. Büttner. (*Chem. Zeit.*, 1913, **37**, 662.)—The official method of the Verein Deutscher Düngerfabrikanten for the analysis of ammonium superphosphates does not prescribe any definite temperature for the aqueous extraction. The effect of temperature, however, is shown in the results obtained by the author. Analyses of two samples containing 5 and 3 per cent. of nitrogen and 10 and 15 per cent. of phosphoric acid (P_2O_5) respectively gave from 0·3 to 0·6 per cent. less phosphoric acid than was anticipated. During the thirty minutes' shaking the temperature had fallen to about 16° C.; by using water at about 35° C., so that the temperature after thirty minutes' shaking did not fall below 18° to 20° C., about 0·5 per cent. more phosphoric acid was obtained. Somewhat higher results were also obtained by passing the sample through a sieve of 1 mm. mesh.

Note by Abstractor.—It is not stated whether the temperature has the same influence in the analysis of other superphosphates. C. A. M.

Estimation of Polythionate in Presence of Thiosulphate and Free Sulphurous Acid. W. Feld. (*Zeitsch. angew. Chem.*, 1913, **26**, 286-288.)—The method previously described by the author (*Zeitsch. angew. Chem.*, 1911, **24**, 290) gives accurate results when the amount of sulphur dioxide in the solution is very small. But when solutions containing notable amounts of sulphur dioxide are boiled with mercuric chloride, a large part of the sulphur dioxide escapes before the oxidation is complete. The following modification of the method gives accurate results even in presence of large proportions of sulphur dioxide. It depends on the fixation of sulphur dioxide as manganous thiosulphate by treatment with excess of manganous sulphide. On subsequent treatment with mercuric chloride, the manganous thiosulphate liberates hydrochloric acid: $3SO_2 = 2MnS_2O_3 = 4HCl$. When treated with mercuric chloride, a molecule of ammonium thiosulphate, like manganous thiosulphate, liberates two molecules of hydrochloric acid, whereas ammonium tetrathionate liberates twice as much acid. On these reactions and the known behaviour of sulphur dioxide and thiosulphates towards iodine, the new method is based.

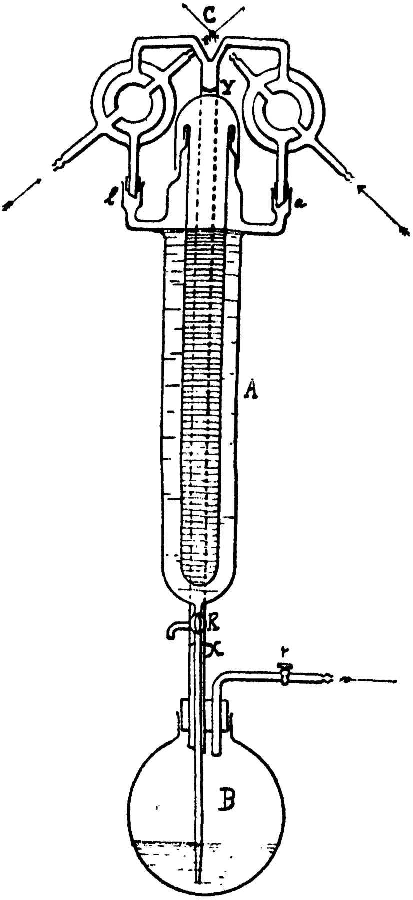
A measured portion of the solution is first titrated with iodine, and then, after addition of ammonium chloride, with caustic alkali of the same normality as the iodine solution, using methyl orange as indicator. Half the consumption of alkali is the measure of the sulphur dioxide originally present, and if half the volume of alkali used be subtracted from the volume of iodine consumed in the first titration, the remainder is the measure of the thiosulphate. A separate portion of the original solution is treated in the cold with excess in suspension of manganous sulphide, and the mixture is left for half an hour, with occasional agitation. It is then made up to known volume, filtered, and an aliquot portion of the filtrate is boiled with excess of mercuric chloride, and after addition of ammonium chloride is titrated with caustic alkali. From the amount of acid found, that due to the sulphur dioxide and thiosulphate originally present is deducted, and the remainder is calculated to tetrathionate.

The suspension of manganous sulphide is prepared as follows: 600 c.c. of a $\frac{M}{1}$ solution of manganous sulphate is warmed to 60° C., and treated with hydrogen sulphide to reduce any higher oxides of manganese. When the solution appears to undergo no further change, a few drops of caustic potash solution are added, the current of hydrogen sulphide continued for a few minutes, and the solution is filtered. The filtrate is warmed to 60° C., and hydrogen sulphide is again passed through it, when it should remain clear. The stream of hydrogen sulphide is now made a vigorous one, and a solution of some 56 grms. of caustic potash (not soda) in a little water is added drop by drop. The mixture is next kept at about 80° C. on the water-bath until it no longer smells of hydrogen sulphide, and until the supernatant liquid no longer reacts with iodine. The process may be hastened by leading hydrogen over the surface of the liquid. The mixture is finally diluted to 1,000 c.c., and is then an approximately $\frac{M}{2}$ suspension of manganous sulphide in approximately $\frac{M}{20}$ manganous sulphate solution. It keeps unchanged for many months. G. C. J.

Estimation of Alkali Sulphides. D. McCandlish and J. A. Wilson. (*Collegium*, 1912, 80-84; through *Chem. Zentralbl.*, 1913, I., 1892.)—With regard to the method proposed by Blockey and Mehd (ANALYST, 1912, **37**, 325), the authors point out that the ammonium chloride solution employed is not sufficiently alkaline to prevent loss of hydrogen sulphide due to hydrolysis of sodium sulphide in aqueous solution, and that it does not contain enough ammonia to prevent the zinc from being precipitated as hydroxide by the lime liquor. This difficulty may be overcome by using a solution containing a sufficient but not undue quantity of alkali, and an ammoniacal zinc sulphate solution is recommended containing such an amount of ammonia as will inhibit the precipitation of zinc hydroxide without interfering with the precipitation of the zinc as sulphide by converting the zinc into a complex amino-salt. Concordant results may be obtained in the analysis of sulphide solutions containing varying quantities of calcium hydroxide when ammoniacal zinc sulphate solution is used, whilst ammonium chloride solution gives erratic figures. In the case of sulphide solutions which are stronger than $\frac{N}{60}$, it is recommended that 25 c.c. of the solution be titrated with $\frac{N}{10}$ zinc solution; another portion of 25 c.c. of the sulphide solution is then treated with three-fourths of the quantity of zinc solution required for the first titration, the mixture is diluted to 100 c.c., filtered, and 25 c.c. of the filtrate are titrated; the amount of zinc sulphate solution required for this last titration is multiplied by 4, and the result added to the number of c.c. (three-fourths) introduced previously. W. P. S.

"Salt Error" in Colorimetric Estimation of Hydrogen Ion Concentration of Sea-Water. S. P. L. Sörenesen and S. Palitzsch. (*Biochem. Zeitsch.*, 1913, **51**, 307-314.)—The relation between salt-content of the water and the correction that must be applied to hydrogen ion concentration found colorimetrically is expressed graphically in the form of curves. The correction varies with the indicator used and liquid with which it is compared. Experiments were made with the following combinations: (1) Borate mixture and phenolphthaleïn, (2) borate mixture and naphtholphthaleïn, (3) phosphate mixture and naphtholphthaleïn, (4) phosphate mixture and neutral red. Correction curves are given for the first three; in the case of phosphate mixture and neutral red no correction is necessary if the salt-content is less than 10 per 1,000. E. W.

APPARATUS, ETC.



Dialysing Apparatus. W. Kopaczewski. (*Comptes rend.*, 1913, **156**, 1853-1855.)—The apparatus here shown may be used for the rapid dialysis of, *e.g.*, enzymes into pure running water, the distillation of the water, and the concentration of the dialysable part. The dialyser *A* communicates by means of a two-way tap with the flask *B*, and with an exterior tube for taking samples of the water. The cork of the flask receives three tubes, one of which, *r*, communicates with a vacuum pump, the second with the dialyser, and the third (of tin) with the Soxhlet coolers *C*, which are connected together by means of a tin tube with two arms. In using the apparatus, the flask is charged with a quantity of water equal to that to which it is desired to bring the dialysable part of the liquid. The tap *R* is closed while a vacuum is created within the flask, after which the tap *r* is closed, the liquid then heated to boiling-point, and the dialysis carried out at a temperature of about 40° C. The apparatus may also be used for distillations *in vacuo* without previous dialysis.

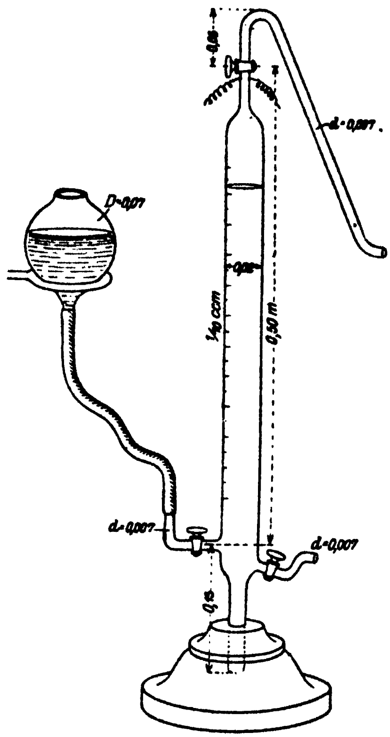
C. A. M.

International Standards for Coloured Fluids, and a Suggested Plan for such Standardisation. H. V. Arny. (*Eighth Inter. Cong. App. Chem.*, 1912, Vol. 26, 319-327.)—The author describes a method of colour-matching in which he uses $\frac{N}{2}$ slightly acidulated (hydrochloric acid) solutions of (red) cobalt chloride (yellow) ferric chloride, and (blue) cupric sulphate, and blends these in any desired proportion. Starting with the possible combinations of the three colours that would make 12 c.c. when fractional portions of the c.c. are not employed, he has prepared a set of eighty-eight blends, the tints of which, tabulated in the paper, range from the pink of cobalt to the blue of copper. He suggests a system of colour nomenclature based on the proportion of the $\frac{N}{2}$ solutions employed to make the tint, and reports at length on such "Co-Fe-Cu" factors for solutions of caramel and cudbear.

H. F. E. H.

Apparatus for Gas Analysis. J. S. Agraz. (*Zeitsch. anal. Chem.*, 1913, **52**, 418-419.)—The apparatus, which is made by the Vereinigte Fabriken für Laboratoriumsbedarf in Berlin, was designed with a view to producing a single piece of apparatus which would serve for a variety of purposes. The illustration is self-explanatory, except that the graduated tube is of 100 c.c. capacity, divided to 0·1 c.c. The dimensions given are fractions of a meter.

G. C. J.



Standardisation of Hydrometers. (*Memorandum issued by the Director of the National Physical Laboratory*, through *Nature*, 1913, **91**, 412-413.)—The Director has issued these notes with a view of eliciting opinions from makers and users, and of obtaining information from other countries, as there is at present much ambiguity as to the bases of standardisation of hydrometers graduated to read direct in degrees of sp. gr. The instruments are in all cases graduated for use in a liquid at a definite temperature, called the standard temperature of the instrument, and give the sp. gr. of this liquid at some definite temperature, which may or may not be the standard temperature of the instrument, referred to water at the same or some other temperature. The following three cases have arisen in practice, and it is desirable to fix upon one of them as the standard method:

I. The water to which the sp. gr. is referred must be at the standard temperature of the instrument.

II. The water to which the sp. gr. is referred must be at some other definite temperature—*e.g.*, 60° F., or possibly 4° C. Thus, if 85° F. is the standard temperature of the instrument, and 60° F. that of the water, the sp. gr. of the liquid at 85° F. is referred to water at 60° F.

III. The graduations are such as to give the value which would be found for the sp. gr. of the liquid if it were cooled or heated to some other temperature and referred to water at that [or IV. some other] temperature. Thus, if the standard temperature of the instrument is 85° F., the instrument would be used at this temperature, but its graduations would be such as to give the sp. gr., which would be found for the liquid if cooled to 60° F., and referred to water at 60° F.

Thus, using a sugar solution at 85° F. of sp. gr. 1·1484 (Method I.), Method II. would show 1·1447, and Method III. 1·1500. Method I. has been the usual practice at Kew, 60° F. being the standard temperature; No. II. has the advantage that the reference temperature of the water is fixed, and gives results in agreement with the usual definition of sp. gr., which assumes a fixed temperature for the water.

H. F. E. H.

Apparatus and Method for Determining Hydrogen Sulphide in Illuminating Gas. E. P. Harding and E. Johnson. (*Eighth Inter. Cong. App. Chem.*, 1912, vol. 25, 673.)—The hydrogen sulphide is absorbed by a solution of cadmium chloride, liberated in a partial vacuum by hydrochloric acid, and titrated with standard iodine. This gives much more accurate results than Tutwiler's method, and agrees well with the gravimetric cadmium chloride method, which is the most accurate at present in use, but is not sufficiently rapid for control work. The method described requires only from seven to ten minutes for a determination. O. E. M.

Apparatus for the Determination of Dissolved Oxygen in Water. G. A. Soper. (*Eighth Inter. Cong. App. Chem.*, 1912, vol. 26, 265.)—The method depends upon the measurement by permanganate of the oxidation caused in ferrous sulphate by the oxygen dissolved in water. A special form of apparatus is described which allows of the addition of the reagents (ferrous sulphate solution, sodium carbonate, and sulphuric acid) without exposing the water to the air. The bottle employed has a capacity of about 500 c.c., and has a long funnel-shaped top rising above the stopper, which is convex at the bottom, and when in place allows about 15 c.c. of liquid to stand in the funnel without overflowing. The capacity of the bottle is accurately gauged with the stopper in position. After filling the bottle with the water to be examined, and emptying the excess which rises in the funnel, 6 c.c. of the standard ferrous sulphate (144 grms. of sulphate and 15 c.c. concentrated sulphuric acid diluted to 3 litres) are delivered by a pipette to the bottom of the bottle. The stopper is replaced, and the water which rises in the funnel is gently poured off. Five c.c. of sodium carbonate solution (20 grms. crystals per 100 c.c.) are then poured into the funnel, and the stopper gently raised sufficiently to allow the heavy alkali to sink through the water to the bottom of the bottle. The stopper is lowered, the excess of water in the funnel is poured off, and the bottle shaken until the free oxygen is entirely absorbed. Ten c.c. of a 50 per cent. solution of sulphuric acid are then added in the same way, and the contents of the bottle are then turned out into an Erlenmeyer flask and titrated with permanganate. A blank determination is made whenever the water contains much organic matter or chlorine, the same process being followed, omitting, however, the addition of sodium carbonate.

H. F. E. H.

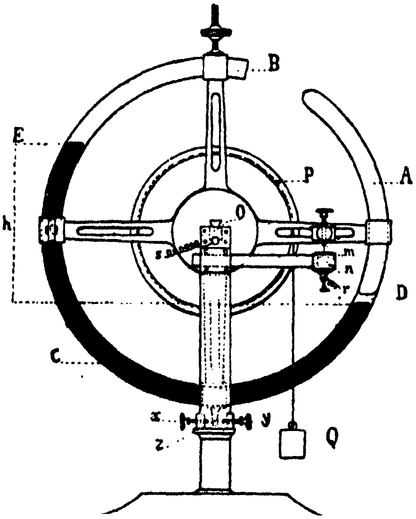
Practical Field Method for the Determination of Dissolved Oxygen in Water. G. A. Soper and P. B. Parsons. (*Eighth Inter. Cong. App. Chem.*, 1912, vol. 26, 267-269.)—The method employed is similar to that in the preceding abstract, the sample being collected in a special form of apparatus, consisting of a separating funnel connected with an empty bottle of much larger capacity. The two are lowered together by a rope to any desired depth, the method of their connection being such that before the bottle can be filled the separating funnel has first to fill and empty itself several times. The separating funnel is then filled with the sample of water in the bottle, and the determination of the dissolved oxygen proceeded with as already described. A control sample which has been collected in a similar manner is also tested, but in this case the sulphuric acid is added first and the whole well shaken before introducing the ferrous sulphate, the acid reaction thus preventing the dissolved oxygen from acting on any of the ferrous salt. The difference between these results is the amount of ferrous salt acted on by the dissolved oxygen of the water. The form of separating funnel is substantially the same as that described by G. A. Letts (Appendix VII., Fifth Report of the Royal Commission on Sewage Disposal). The method is claimed to be especially suitable for field work and for tidal waters, or those containing nitrites. H. F. E. H.

Methods and Apparatus used in Petroleum Testing. Part II.: Viscosimetry. W. F. Higgins. (*J. Soc. Chem. Ind.*, 1913, **32**, 568-572.)—An abstract of a paper which described experiments made at the National Physical Laboratory, the chief object of which appears to have been to determine whether some simple expression could be found connecting true viscosities with the observed times of efflux from a Redwood viscosimeter, and to determine the order of accuracy which would attach to viscosities calculated by the use of such an expression. A comparison is also instituted between the results obtained by Redwood's and Engler's viscosimeters respectively. For a particular instrument of the Redwood type, and with times of efflux ranging from 40 to 2,000 seconds, it was found that the expression $\eta_t = (0{\cdot}0026T - \frac{1{\cdot}715}{T})\,\delta$ gave numbers which differed from the true viscosities by less than ±5 per cent., T being the time of efflux in seconds and δ the density of the liquid at t° C., the temperature at which the experiment is made and at which it is desired to know the true viscosity, η_t. Ubbelohde (*Tabellen zum Englerschen Viskosimeter*, 1907) has arrived at the formula $\eta_t = (0{\cdot}001435\,T - \frac{3{\cdot}22}{T})\,\delta$ for calculating viscosities from the observed times of efflux from Engler's viscosimeter, T in this case being the time of efflux, not of 50 c.c., as used with Redwood's instrument, but of the normal Engler quantity of 200 c.c. From these two equations the author has calculated the ratio T_E/T_R (time of efflux with Engler's viscosimeter / time of efflux with Redwood's viscosimeter) for nine values of T_R. For values of T_R from 5,000 down to 150, this ratio is almost a constant, 1·81. As T_R is further diminished, it increases slightly, becoming 1·83 when $T_R = 40$. Experimental comparison of an Engler and a Redwood instrument pointed to a ratio $R_E/R_T = 1{\cdot}8 \pm 0{\cdot}06$. The same series of experiments showed that Ubbelohde's formula or tables, when used to calculate viscosities from the results of experiments with the particular Engler viscosimeter possessed by the author, gave numbers mostly above the truth, the errors ranging from − 2 per cent. to + 8 per cent. G. C. J.

Note by Abstractor.—The paper as printed contains two errors clearly attributable to its having been curtailed for purposes of publication, and the printed text would justify the assumption that the author was putting forward his formula as applicable to all Redwood viscosimeters. This would conflict with the universal experience that the indications of these instruments may vary, and the printed discussion shows that the author is aware of such variations. The full text of the paper may be expected to appear in the next volume of "Collected Researches of the National Physical Laboratory," or in some publication of the International Petroleum Commission, on whose behalf the experiments were made.

Apparatus for Recovery of Evaporated Organic Solvents. W. Friese. (*Pharm. Zentralhalle*, 1913, **54**, 419; through *Chem. Zentralbl.*, 1913, I., 2015.)—The apparatus consists of an inverted double-walled, water-cooled funnel standing on three feet in a glass or metal circular trough, which fits over the evaporating basin, and has a side tube through which the recovered solvent runs off. It is made by Hugershoff, Leipzig. O. E. M.

New Thermo-Regulator. E. Esclangon. (*Comptes rend.*, 1913, **156**, 1667-1670.)—This consists of a circular tube, *A C B*, closed at *A* and open at *B*. It revolves round its centre, *O*, and at *P* carries a pulley, from which is suspended a weight, *Q*. The closed part of the tube, *A*, contains a saturating vapour, the nature of which depends upon the temperature which the apparatus is required to maintain. Thus for temperatures between 35° and 40° C. ether is a suitable substance, for temperatures about 55° C. acetone may be used, and for temperatures about 100° C. water is suitable. The centre of gravity of the apparatus is on the axis *O*. Any alteration in the temperature will destroy the equilibrium and cause movements of the apparatus, the extent of which is controlled by the stops *x* and *y*. All that is required is to connect the movements with a heating or cooling apparatus. C. A. M.



Comparison of Redwood's and Engler's Viscosimeters. W. Meissner. (*Chem. Rev. Fett Ind.*, 1913, **20**, 123-126.)—Practical tests with standard instruments have been made, and the results compared with those calculated by means of the author's formulæ (Analyst, 1912, **37**, 223). The relationship between the two viscosimeters was found to vary with the temperature. The ratio between the Redwood values and the Engler degrees, which at 20° C. deviated from unity by amounts up to 11 per cent., was found to be about 6 per cent. greater at 50° C. than at 20° C. The explanation is that at 50° C. there are differences in temperature between the different parts of the apparatus, but it has not yet been ascertained in which type of viscosimeter the influence of these differences is the greater.

C. A. M.

Precision Viscosimeter for Measurement of Relative Viscosity and the Relative Viscosities of Water at 0°, 18°, 25° and 50° C. E. W. Washburn and G. Y. Williams. (*J. Amer. Chem. Soc.*, 1913, **35**, 737-750.)—An improved viscosimeter of the Ostwald type is described. Its water-constant at a given temperature is not changed by cleaning with hot mixtures or by subjecting the viscosimeter to large variations of temperature. The water-constant (about 580 seconds at 25° C.) can be repeatedly reproduced to about 0·03 second under a given set of conditions. For effective pressures within the limits 130 and 300 mm. of water the deviations of the viscosimeter from Pouseuille's law is less than 0·03 per cent. The relatively large size and flattened shape of the lower bulb of the viscosimeter have the effect of reducing to negligible proportions any error due to small inequalities in the volume of liquid taken for the test. The normal charge with this instrument is 65 c.c., and an error of 1 c.c. in the measurement has so little effect on the mean head of liquid during the experimental interval that the time of flow varies no more than 0·05 per cent. A single instrument of this type can be used for a large temperature range, because its dimensions do not vary with varying temperature. The quartz viscosimeter is made by the Silica Syndicate, London.

The relative viscosities of water at 0°, 18°, 25°, and 50° C. are given as $\eta_{18}/\eta_0 = 0{\cdot}58978$, $\eta_{25}/\eta_0 = 0{\cdot}49741$, and $\eta_{50}/\eta_0 = 0{\cdot}30640$, with a probable error of ± 0·03 per cent. In obtaining these results the authors made use of chronometric apparatus permitting periods of time up to one hour to be recorded within 0·01 second, and of thermostatic appliances permitting temperature regulation to within a few thousandths of 1° C. These appliances are described in the paper, which also includes a statement of the reasons which determined the choice of the exact form and dimensions of the viscosimeter.

G. C. J.

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REVIEWS.

Industrial and Manufacturing Chemistry: Organic. By Geoffrey Martin, Ph.D., M.Sc. (and Others). Pp. xx+726. London: Crosby Lockwood and Son. 1912. Price 21s. net.

To review a book of this cyclopædic character fairly is always difficult, especially so when, as in the present case, the reviewer comes from his perusal of it with a more lively sense of its shortcomings than of its merits. The first question which will be asked concerning such a book is, For whom does it cater or set out to cater, and what is its scope? Let the preface speak for itself:

"This book is a treatise on the applications of organic chemistry to the arts and manufactures. It embraces both British and American practice, and affords, so far as is ascertainable in view of the many secret processes employed, thoroughly up-to-date information regarding the various branches of chemical industry. . . . The editor's aim has been to cover the whole range of subjects with which the industrial chemist and manfacturer are usually concerned, and the book will serve either as a textbook or as a work of reference; it is intended to meet the requirements of all business and practical men interested in chemical processes, of manufacturers, consulting chemists, chemical engineers, patent workers, inventors, technical lawyers, lecturers on technology, fire-insurance inspectors, and others."

Any chemist reading the above will appreciate the reviewer's difficulty, especially if one or more subsections of the work are useful contributions to chemical literature, for the book as a whole must fail to achieve the editor's aims. Thorpe's "Dictionary of Applied Chemistry" even achieves less, and the editor of that work did not attempt the almost impossible task of providing matter which should be at once intelligible to insurance inspectors and technical lawyers, and yet instructive to chemical engineers and manufacturers.

The book is divided into twenty-three sections, and the labour of producing it has been very unequally shared by fifteen contributors. As the sections are still further subdivided (Section I., for example, into thirteen subsections, the work of five writers), no reviewer can be expected to indicate the relative value of each fragment of the text, nor could a reviewer be found competent to deal with "the whole range of subjects with which the industrial chemist is concerned." All that can be attempted is to examine the book as to its possible usefulness to any of the varied types of person to whom it is addressed.

Readers of this journal will wish to know if the book is likely to afford any help to the analyst faced with a proposition unfamiliar to him. Now, in fairness to the contributors, it must be pointed out that the book makes no pretence to be a textbook of analytical methods. But a description of analytical methods is a prominent feature of the book, and one has a right to expect such information and references as are given to be accurate. On pp. 263-264 we are told that "the percentage of extract in malt is usually determined in the laboratory by first grinding it into the finest possible powder, and then extracting with a definite quantity of water at 60 to 65° C., and taking the rotation of the filtered solution with the polarimeter." For details of this method we are referred to Bailey's "Brewers' Analyst." Now Bailey's "Brewers' Analyst" will not bear the most casual scrutiny of a chemist, and no one with any knowledge of the work of a brewing laboratory could have been guilty of the paragraph quoted. The section on brewing which is by the editor and Mr. C. H. Griffiths, a brewing engineer, contains far too many mistakes to be a safe work of reference: the real technologist will learn nothing, and other readers may be misled. The name of Dr. A. Slator, who is an authority on brewing chemistry, appears on the title-page, but his sole contribution is a two-page article on the rate of alcoholic fermentation.

As this book claims to embrace both British and American practice, we put ourselves in the position of a British manufacturer seeking information on an industry which has attained to an exceptional state of development in America—the maize starch industry. We found eight lines on p. 178. The preface says that particular pains have been taken to incorporate minor industries, as such minor industries, through absence of competition, frequently afford profits far greater than those of fully developed trades. It is within the reviewer's knowledge that the apparent gross profit to be made out of caramel has tempted many persons to inquire into this industry. In the index to this book such a person will find "Caramel, 142, 176, 265," and on turning to these pages will learn that sucrose on heating loses water, yielding caramel; that Germany produces from starch about 5,000 tons of caramel or burnt sugar; and that, in order to obtain dark beer and produce a pronounced malt flavour, caramel and black malt are added in small amount, mixed with the other brewing materials. Rather a poor reward for turning up three references!

The above experience directed your reviewer's attention to the index, which occupies no less than sixty pages—surely a good point in a work of reference, if quality matches quantity. To test its quality, "acetone" was selected at random. Apart from sub-indexing, under "recovery" and other words, no less than ten page references follow the word "acetone." Of these, three are in heavy type, and a note informs us that "When several references are given, the ones with heavier type denote the more important." Will it be believed that one of the three references is to a page dealing with the synthesis of the eucaines? Further experiment with the index confirmed the impression that it was the work of someone unfamiliar with the subject-matter. Such a person might be excused for the heavy-type reference to the page relating to the synthesis of the eucaines, for that page not only contains the word "acetone" several times, but is embellished with the graphic formula of that substance duly labelled. Relegating the indexing of a technical book to an incompetent person, however, is inexcusable. It may waste incalculable hours if the book gets a circulation. Anyone looking up "tartar emetic," for instance, would turn to p. 583, glance at it, suspect a misprint, try p. 383, or consult the twenty-page table of contents, and then give it up. There is no misprint. The only reference in the book to tartar emetic is the statement that it is used as a mordant in dyeing leather, and this appears on p. 583. The omission of any further reference to tartar emetic is unimportant, but the name of the substance has no right to appear in the index at all.

Many of the articles in the book are carefully written, the names of the contributors being almost sufficient guarantee of this. For example, the section on leather, by D. J. Law, appears to be an excellent cyclopædia article. Mr. Law does not attempt to cater for the varied clientèle to which the editor appeals in his preface. He has neither tried to impress expert critics with his erudition, nor written down to the level of the fire-insurance inspector. Consequently, a chemist or engineer with little or no knowledge of the leather industry, suddenly compelled to acquire a broad acquaintance with some branch of it, will probably find the section helpful, and will certainly not be misled. Mr. C. A. Mitchell's name is a guarantee of the care with which some other sections have been written, but it is difficult to understand why he was asked to write a three-page article on essential oils. No one will doubt Mr. Mitchell's capacity to write a book on the subject, but in three pages what can he tell which is not to be found in one or more books on the shelves of every chemist, even those whose work seldom or never brings them up against essential oils? As the section is scarcely intelligible to the non-chemist, it is difficult to see what useful purpose it can serve.

As has been said already, your reviewer cannot take a book of this kind section by section and say which are trustworthy. In the sections dealing with the industries with which he is most familiar he finds so many inaccurate statements that he would fear to trust to other sections by the same writers for information on subjects less familiar to him. His duty would therefore seem to be to warn others against relying on the book. The illustrations deserve a word. It is often necessary for a chemist or engineer unfamiliar with a particular industry to make himself acquainted with the general features and lay-out of the plant employed in that industry. Often he can find few illustrations, and those antiquated. The illustrations in this book are numerous, and almost all of them reproduced from current makers' catalogues.

G. CECIL JONES.

BEURRES ET GRAISSES ANIMALES. Par ALBERT BRUNO. (MANUELS PRATIQUES D'ANALYSES CHIMIQUES.) Paris: Ch. Béranger. 1912. Price 6 f.

This manual, one of the series of practical treatises on chemical analysis, published under the direction of Mons. Bordas and Roux for use in official laboratories and elsewhere, is chiefly concerned with French official methods for the examination of butter, animal fats, and margarine.

The methods set forth are almost entirely of an official character and consequently somewhat ponderous, as such methods usually are. They are, however, well and carefully described, but being intended principally for French official practice, they are not likely to be of great use to the English chemist, who will find more practical and more modern methods in the literature of his own country. Still they have the advantage of showing him how results are obtained in French official practice, and their value is enhanced by a most useful chapter on "International Notation," and the methods by which results as usually obtained may be converted into this form of expression.

The introduction contains some very sound remarks on the confusion that arises from the employment of varying forms of expressing analytical results, and the author expresses the wish (which will be echoed by many) that certain standard methods might be adhered to in official work, and a stop be put to the unceasing stream of new and often doubtful methods which swells the already large literature of this subject.

The chapter on expression and interpretation of results is distinctly good and is illustrated by many useful tables founded on the methods described, but the author rightly remarks that figures obtained by the actual investigator are of more use to him than published figures.

At least half the volume is devoted to butter, and the sections dealing with lard, margarine, and tallow are consequently very condensed; and whilst the details of examination given are sound and practical, they will not be of much use to those who are not fairly well acquainted with the subject, and might well have been treated more fully.

The book, whilst in every way excellent, is not suitable for those who desire guidance in the occasional examination of butter, etc. At the same time, those whose work lies principally in this direction will probably find much to interest them.

The various laws and regulations concerning these products, which are and have been in force in France are fully set out, together with a synopsis of the regulations in force in practically every civilised country. These appendices will be of considerable value to those who have constantly to examine the produce of other countries, and it is to such, as has been said, that the work will be of chief interest.

CECIL REVIS.

THE QUALITATIVE ANALYSIS OF MEDICINAL PREPARATIONS. By H. C. FULLER. New York: John Wiley and Sons; London: Chapman and Hall, Ltd. 1912. Pp. ix + 132. Price 6s. 6d. net.

Public Analysts and other analytical chemists who, from following the proceedings of the Select Committee of the House of Commons on Patent and Proprietary Medicines, or from other causes, have realised the enormous difficulty of analysing, even qualitatively, complicated mixtures of vegetable extracts and other preparations of drugs, will be inclined to turn hopefully to any new work promising assistance in the analysis of medicinal preparations. In the case of the small volume under review disappointment will follow hope.

The book is by an American writer for American readers, and relates apparently to American Patent Medicines. It is a vast collection of "facts" largely relating to bodies many of which are but rarely met with, in this country, at all events. Alphozone, Chinaphenin, Coronillin, Oxaphor, Periplocin, and Spirosal, are half a dozen selected at random.

Among the long list of substances enumerated, the occurrence of many of which must be rare, the limit of unlikelihood surely being reached with skatol, the reviewer failed to find mention of several commonly occurring and important medicinal chemicals, as, for instance, formates and glycerophosphates. Throughout the classified lists of substances where italics are used to call attention to the commoner substances, the use of italics does not appear to have been made with much discretion.

The author's style is abbreviated to breaking-point, and frequently leaves something to be desired, as instance the statement on p. 3, that lead acetate is "*partly* soluble in alcohol," and this sentence on p. 86: "Be sure the lime and magnesia are free from arsenic, or decompose with nitric and sulphuric acids." Throughout the work the names of alkaloids are denied the terminal "e," which is irritating to English readers, while split infinitives and errors of syntax are not wanting.

The first portion of the book (pp. 1 to 78) is concerned with the scheme of analysis for the separation and detection of substances in medicinal products, and consists largely of long lists of such "substances." Crude drugs, resins, extracts, oils, and other mixtures are designated "substances" in this book. The tests given for identifying the "substances" are very largely colour reactions. In some cases melting-points are given; but information as to how the "substances" are to be isolated in order that the colour reaction may be applied, or the melting-point determined, is not forthcoming. Thus, on p. 86, the reader is instructed to "remove a portion and test for sucrose, citrates, tartrates, acetates, albuminous material, hypophosphites, hypochlorites, peroxides, and for substances which may become altered by heat," without any guidance as to how these operations are to be performed.

It is well known that colour reactions are only to be relied upon when the substance is free from admixture, such as usually occurs, unless great precautions are taken, in the case of separation from complicated organic mixtures. This interference the author recognises in the Introduction, but not in the body of the work, except that on p. 35 he frankly remarks that "the colour reactions given with sulphuric acid are usually only characteristic when the substances are pure and alone." Similarly, in order that the melting-point may be used as an analytical factor, the substance—which may be a minute quantity of an alkaloid or glucoside—has to be separated and purified, an operation frequently of extreme difficulty.

Aconitine is ineptly stated on p. 36 to melt at "182° to 186° slow heating." Now, the melting-point of aconitine varies according to the rate of heating, the alkaloid being decomposed by slow heating; but by no means can a distinctive melting-point be obtained within the range given in this book. On p. 56 a test is given for aconitine which deserves strong condemnation, the reader being instructed as follows:

> "*Aconitin.*—Dissolve residue in water containing a few drops of acetic acid and take 1 to 2 c.c. in the mouth, rolling it round with the tongue, and then expectorate. If aconitin is present, the tingling sensation will soon appear. If sufficient alkaloid is present, prepare the aurochloride," etc.

For the personal safety of some readers it seems expedient to remark that, if sufficient aconitine is present, there would hardly be time to prepare the aurochloride before death supervenes, even though the analyst exhibit transatlantic facility in expectorating. The author continues:

> "If sufficient alkaloid is present, prepare the aurochloride, which is a well-defined crystalline salt, wash, dry, and take the melting-point, which, in the case of the pure salt, is 135°."

Inasmuch as aconitine aurochloride exists in three modifications, each having its distinct melting-point, these instructions are scarcely adequate for the identification of aconitin.

In fact, the treatment of the alkaloids cannot be said to be at all satisfactory. No reliable instructions, for instance, are detailed for the separation and identification of so important an alkaloid as morphine, nor, indeed, for any other alkaloid.

Among such tests, other than colour reactions and melting-points, as are to be found, there is a lack of thoroughness and accuracy which prevents the acceptance of this book as a safe analytical guide. Thus, on p. 15, the development of a blue colour with ferric chloride as proof of the presence of epicarin, even when salicylic acid and vanillin have been excluded, appears to lack finality. On the same page reduction of ammoniacal silver nitrate is stated to indicate either chloral or dormiol;

and a subtle colour test for subcutin is applied to a residue regardless of the interference of other substances. On p. 24 aspirin is stated to give a violet coloration with ferric chloride, a result which is not obtained unless the acetylsalicylic acid (aspirin) contains an impurity—*e.g.*, salicylic acid. On pp. 78 and 79 there is detailed a procedure for examining preparations for arsenic which is superfluous to the expert analyst, and an unsafe guide for others; while on p. 25 the author himself apparently tires of the long list of tests, for he naïvely remarks: "If this scheme does not serve to identify the substances present, proceed according to Mulliken's scheme for the identification of organic bodies."

Pp. 81 to 108 are devoted to methods of analysis of certain classes of pharmaceutical products. Of these special sections that on "Elixirs" is probably the best, the subject being treated systematically and in such a manner as to be of real use to non-pharmaceutical analysts. One regrets that other sections in the book are not treated on similar lines. The "Extracts" form one of the most important classes of pharmaceutical preparations, and their analysis presents, perhaps, the greatest difficulty; yet this is at once one of the briefest and weakest sections in the book. One looks in vain for mention of such common and important extracts as those of ergot, opium, nux vomica, belladonna, Indian hemp, liquorice, senna, cinchona, rhubarb, and male fern. Under this section, on p. 93, the author's naïveté once more asserts itself and prompts him to state (of extracts) that "they often take part in the preparation of pharmaceuticals, but alone one is seldom called to analyse them, and when they are sold in this way they are usually labelled so that an identification is not difficult."

Ointments, on p. 102, are stated to contain, besides "alkaloidal bodies dissolved as oleates in suspension," also "flavouring agents," which excites one's curiosity as to whether in the author's view ointments merit the same treatment as aconitine.

Pills are stated to contain gypsum (an unlikely constituent), but no mention of French chalk, which forms the coating of "pearl-coated" pills, is to be found.

To systematise the qualitative analysis of drugs and "substances" used in medicine is a task of such extreme difficulty as to be virtually an impossibility, and to say that the author has not succeeded is not to deny utility to his book. To work out a qualitative analysis of even the mixtures and compounds mentioned in this book would occupy a lifetime, and it may be said with little fear of contradiction that a life could easily be better and more profitably spent. C. A. HILL.

CHLORIDE OF LIME IN SANITATION. By ALBERT H. HOOKER. New York: John Wiley and Sons. 1913. Pp. v+231. Price 12s. 6d. net.

There is little matter of analytical interest in this book, but there is a large amount of information which will be useful to chemists engaged in the control of water and sewage purification, and in disinfection work generally.

The book is divided into two distinct parts. The first one-third of the book consists of an account of the use which has been made of chloride of lime in the sterilisation of drinking-water, in the treatment of sewage, in the watering of streets, in surgical practice, on the farm, and in the destruction of house-flies. These chapters are written in a style which can be followed by the ordinary reader, as it appears to be part of the author's object to encourage the more general use of disinfection processes. The following two-thirds of the book is the more important part to the technical reader. It consists of a bibliography of the literature of the matters treated in the first part, extending to 442 entries, and classified according to subjects. In nearly all cases an abstract of the reference quoted is given. Many of these abstracts are those of official and annual reports, the contents of which frequently fail to become generally known; and for thus drawing attention to them the author deserves the unstinted thanks of workers in the subject. There is an index to subjects and authors, and the names of the authors mentioned in the bibliography are included.

J. H. Johnston.

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INSTITUTE OF CHEMISTRY.

PASS LIST: JUNE—JULY (1913) EXAMINATIONS.

London and Glasgow.

Of thirty-three candidates who presented themselves for the Intermediate Examination, sixteen passed: R. L. Amoore, R. O. Bishop, A. Dingwall, J. W. Donaldson, B.Sc. (Edin.), J. G. Duncan, A. Dunsmore, J. S. Frith, Peter Kerr, B.Sc. (Edin.), K. G. Lochhead, J. W. Lorimer, H. V. Parker, B.A. (Cantab.), H. C. Reynard, F. Smith, F. W. Snelgrove, B.Sc. (Lond.), A. R. Steele, and A. F. Weiss, B.Sc. (Lond.). Of forty-five candidates who presented themselves for the final examination, twenty-four passed; in the Branch of Mineral Chemistry: B. Campbell, B.Sc. (Lond.), J. A. Pickard, B.Sc. (Lond.), A.R.C.S. (Lond.), E. A. Rayner, B.Sc. (Lond.), and E. W. Skelton, B.Sc. (Lond.); in the Branch of Metallurgical Chemistry: R. J. Dunn, B.Sc. (Birm.); in the Branch of Physical Chemistry: (Miss) G. Thompson, B.Sc. (Lond.); in the Branch of Organic Chemistry: J. S. Bainbridge, B.Sc. (Leeds), A. L. R. Clarke, B.Sc. (Lond.), A. Cunningham, B. B. Dey, M.Sc. (Calcutta), J. R. Gray, G. N. Grinling, E. S. Hawkins, B.Sc. (Birm.), P. C. R. Kingscott, A.R.C.S. (Lond.), D. E. Sharp, B.Sc. (Aberdeen), T. F. Smeaton, E. W. Smith, B.Sc. (Lond.), and R. Wheatley, B.Sc. (Leeds); in the Branch of the Chemistry of Food and Drugs, and of Water: (Miss) D. J. Bartlett H. B. Brown, D. W. Kent-Jones, B.Sc. (Lond.), H. A. Phillips, H. V. Potter, and S. H. Stroud.

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THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

THE USE OF GLYCERIN-JELLY IN MOUNTING MICROSCOPICAL OBJECTS.

By L. W. STANSELL, F.I.C.

(*Read at the Meeting, April* 2, 1913.)

Mounting with this medium is quite simple if the right course is followed. It is a process specially advocated for the laboratory to enable the analyst to preserve for future reference examples of feeding stuffs, jam materials, water sediments, etc. The difficulties usually met with through the introduction of air-bubbles have been overcome, and most of the trouble from this cause averted.

Glycerin-jelly, originally suggested by De la Rue, is dilute glycerol (1 : 1) mixed with about 5 per cent. of gelatine. It can be readily prepared from the standard formulæ of Carpenter or Martindale.

The real secret is to avoid any bubbles of air making their appearance from the outset, and if the process is carried out as directed, glycerine-jelly is a suitable medium to work with for nearly all structures of a vegetable nature.

I proceed as follows: The jelly in the stock bottle is allowed to become liquefied by standing in the water-oven; should it contain air-bubbles, time must be given for these to rise and escape. A few clean 3 inches by 1 inch glass slips are ready upon a flat bench; then, with a glass rod, 1 drop of the limpid jelly is carefully placed on the centre of each slide, the drop not being allowed to fall from the glass rod, but almost *placed* in position.

The drop of jelly soon sets to a clear, lens-like form, usually free from air-bubbles. Should one, however, be seen, it can be dispersed with a warm needle, or else by the same means drawn to one side and pushed out of the way before the jelly sets. These prepared slides are ready for immediate use, or if preferred they can be stored in a box out of the dust for some days until wanted.

It is important to have objects for mounting in the moist condition; particles of fibre from feeding-stuffs will already be in that state if the material has been prepared for the determination of cellulose; they can be taken direct out of the final washing water.

Portions of dry leaves, mosses, and similar structures, are soaked for some days in a preservative fluid consisting of distilled water 2 parts, rectified spirit 1 part, and glycerol 1 part. Each specimen is kept in a small bottle or tube with a little of the fluid; it gradually penetrates and will displace the air, more speedily if put in a warm place.

Some tissues previous to this soaking may even be boiled in distilled water and allowed to remain there till cold. The air-free water helps to dissolve out residual air.

A simple method of preparation to employ for leaves—tea, for instance—is to raise cautiously to boiling in a test-tube with dilute nitric acid, and then freely dilute with water. If the structures float owing to imprisoned air, further boiling in water is necessary before proceeding to wash. The cuticle separates and can easily be stripped off.

The fragments of husk, cuticle, etc., are taken while wet and gently laid with forceps upon the flat bead of solid jelly, the excess of water or other fluid allowed to drain off for a moment, and then the slide slowly warmed over a very small flame. The jelly melts, and the object *in situ* will sink into the liquid jelly without as a rule the introduction of a single air-bubble. The cover-glass is applied and gradually lowered, placing a flattened bullet on it to keep it in position, then left for some minutes till the slide has set. Not much pressure should be exerted, or the cover-glass may have a tendency to spring up afterwards.

At this stage the mount admits of microscopical examination. When this is completed, the processes of finishing the slide can be proceeded with, or if desired, held over, and the slide reserved for treatment with others.

The first step will be to remove with a penknife the excess of solid jelly around the rim of the cover-glass, and to clean everything up with a camel-hair brush repeatedly dipped in water. It is the safer plan to add a brass wire clip so as to guard against the cover moving. The slide is dried with soft rag, and is ready for placing on the turn-table. When centred, a ring of Japanner's gold size is spun round, the purpose of this being to act as a protective coating round the edge of the cover, and to more firmly attach it to the slide. Later on, when dry, a circle of white zinc cement is added perfectly concentric, and after this has hardened, a second further ring coincident with the former. Lastly, when some hours have elapsed, a final touch is given by neatly ringing a narrow line in black Japan or asphalt varnish in the middle of the previously laid white ring.

Many slides can be consecutively finished off together. An artist's small sable for oil-painting is the best brush for the work. It should be cleaned after use each time with turpentine. It is necessary to protect materials and slides in process of finishing from the dust if a perfect appearance in the completed mount is expected.

Sediments and other finely divided objects need different treatment. When mounting the deposit obtained from a water, or the washed residue from Demerara sugar, a conical settling-glass is provided, and when all suspended matters have settled, as much as possible of the supernatant fluid is decanted off until only a drop or two remains. The drop that is left, containing the residue of suspended matter, is poured over the bead of glycerin-jelly already solidified on a slide. By now placing this slide overnight in a level position, in a desiccator, the water will be found next day to have become absorbed, leaving the sediment in position on the surface of the jelly. If now gently warmed and a cover-glass adjusted, any fine residue of suspended matter from water can be more successfully retained and fixed by such means than the usual methods of treatment allow.

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THE RECOVERY OF IODINE FROM RESIDUES.

By HAROLD W. GILL, B.Sc., A.I.C.

(*Read at the Meeting, May* 7, 1913.)

Various attempts have been made from time to time to work out a method for the recovery of iodine in some usable form from the waste solutions obtained in iodimetric analysis. The following method, which depends on the solubility of sodium iodide in absolute alcohol, has been successfully employed:

If in the preparation of standard iodine solutions the iodine be dissolved in sodium iodide solution instead of potassium iodide, as is more usual, the whole of the iodine present can be recovered as sodium iodide by the following method: The residues are evaporated to dryness and heated in an air oven until perfectly dry. They are then coarsely crushed, transferred to a Soxhlet (the lower part of which has been lightly plugged with glass wool), and extracted with absolute ethyl alcohol.

All other salts present in these residues are insoluble in absolute ethyl alcohol, hence the product obtained after extraction is sufficiently pure for many purposes. Recrystallisation from alcohol is sometimes advantageous, but in all cases very careful drying, first in an air oven and afterwards *in vacuo* to remove traces of alcohol, is necessary.

The recovered salt is only slightly deliquescent.

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NOTE ON THE ESTIMATION OF CHLORINE IN RAG FLOCK.

By J. W. BLACK.

The Rag Flock Act of 1911 has fixed a standard of cleanliness for this material, based upon the amount of chlorine in the form of soluble chlorides which can be removed from the flock by thorough washing with water at not more than 25° C., and from not less than 40 grms. of the sample. The examination of a sample of flock taken under this Act necessitates a determination of the amount of chlorine which can be extracted, an operation apparently so simple as to lead one to expect close agreement in the results obtained by different analysts. Legal reports in Trade Journals, however, indicate that this agreement is frequently lacking. In some instances the results show serious discrepancies. Personal information from different analysts who have examined a large number of samples proves that, apart from the question of efficient sampling, there must exist a disturbing influence, either in the method chosen or in the procedure adopted, which causes these discrepancies. In view of this fact, I felt that the results of my experiments with the object of deciding upon a method at once accurate and expeditious might be of some interest.

I first determined the chlorine in a large number of samples by three methods: (1) Gravimetric; (2) direct titration, using potassium chromate as indicator; (3) indirect titration (Volhard's). In this series of tests, the extraction of the flock was made in a 6-inch Buchner funnel fitted to a 58 oz. filtration flask. To maintain the water in the funnel the side tube of the flask was fitted with a rubber clip attachment. Forty grms. of the sample were placed in the funnel and repeatedly wetted with water until the mass could be pressed down as a thick felt. The side tube was then closed and the funnel filled with water. After standing some time, the clip was released and the water allowed to percolate into the flask. This operation was repeated until the volume of the washings was nearly 2,000 c.c., the felt being pressed down as dry as possible. The volume of liquid was then made up to 2,000 c.c., and 1,000 c.c. taken for the gravimetric estimation and 500 c.c. each for the volumetric methods. On evaporating these solutions to workable volumes, I at first experienced considerable difficulty in obtaining solutions suitable either for gravimetric or volumetric estimation, owing to their pronounced colour and the presence of a considerable quantity of flocculent matter which separated out. Dr. Garret (Local Government Board on Chemical Analysis of Rag Flock) observes that in solutions so obtained the colour is almost always too deep for volumetric methods to be employed conveniently. I have not found one sample of flock which yielded an extract suitable for titration without further treatment, nor do I consider that extracts thus prepared are wholly suitable for gravimetric estimation. Filtration was invariably resorted to, and even then the colour of the filtrate was so intense as to obscure the end-point in the direct titration. I found that if a few drops of Mercks' perhydrol were added to the solution before evaporation, the colour was discharged, and a solution obtained which could invariably be titrated without filtration by Volhard's method, and, with few exceptions, by the direct titration. A satisfactory gravimetric test even after the addition of perhydrol could only be obtained by filtration which yielded a clear liquid varying from light straw-yellow to water-white.

The following results were obtained from flocks treated as described (the titrations were performed on 500 c.c. of the extract after evaporation to 100 c.c.):

TABLE I.

Chlorine in Parts per 100,000.

Flock.	Gravimetric.	Volhard's.	Direct Titration.
1	12·37	12·5	12·5
2	14·30	14·0	15·0
3	38·98	42·0	43·0
4	4·94	5·0	5·0
5	20·87	23·0	25·0
6	76·57	76·0	77·0

In the chromate method I applied a correction for the volume of silver nitrate used without producing a visible effect. Before the colour of silver chromate was distinctive 100 c.c. distilled water required 0·15 c.c. of the silver nitrate solution.

The method of extraction followed in the above experiments necessitated considerable time, and, with the view of determining whether this part of the process could be made more expeditious without impairing its accuracy, I made a series of experiments, from the results of which the following modified process of extraction was found satisfactory:

Forty grms. of the flock were put into 500 c.c. of water contained in a large beaker, and the pulp allowed to stand for some hours, preferably overnight. It was then transferred to the Buchner funnel, and thoroughly pressed to a fine felt. Two hundred and fifty c.c. of water were then poured in, and after a few minutes' extraction the mass was again thoroughly pressed. This operation was repeated, and the washing made up to 1,000 c.c. The results obtained by this method of extraction compared with the extraction using 2,000 c.c. of water are shown in the following table:

TABLE II.

Chlorine in Parts per 100,000.

Flock.	Obtained by using 2,000 c.c. of Water.	Obtained by using 1,000 c.c. of Water.
1	14	15
2	5	5
3	23	23
4	76	76

Experiments proved that, to insure a complete extraction of soluble chlorides from 40 grms. or more of flock, a process of percolation and subsequent pressing is necessary. Even when 40 grms. of well-pressed flock are capable of retaining 130 to 150 c.c. of water, and whilst doubtless all the chlorides are rendered soluble by the soaking of the flock in water, it is quite possible that the concentration of the solution is unequal in different parts. It is conceivable that the 150 c.c. retained by the flock may represent a more concentrated solution than the filtered portion. Additional washing of the flock with fresh water obviates this source of error.

I have previously referred to the unsuitability of the directly evaporated extracts for gravimetric estimation. The silver chloride precipitated from such extracts was not only itself invariably highly coloured, but was contaminated with a comparatively large quantity of flocculent matter, which causes the gravimetric results to be higher than those obtained after the decomposition of the dye-stuffs with perhydrol.

In investigating the influence of dye-stuffs on the composition of the precipitate obtained by the addition of silver nitrate, I experimented with flocks yielding intensely coloured extracts of various hues to ascertain if these dyes exerted a progressive reduction of silver nitrate to silver in presence of dilute nitric acid, a reaction which, if not probable, cannot be regarded as inconceivable (Gutzeit's Test). Experiments extending over ten days, however, showed that after the first initial precipitation there was no further reduction of the silver nitrate, although much dye remained in solution. For the purpose of inquiring into the nature of the precipitate a quantity of the impure silver precipitates was collected, carefully guarding against the action of light, and washed thoroughly with cold and hot distilled water. A considerable quantity of dye was removed by the washing with hot water; the washing was continued till the filtrate was colourless and free from silver. On washing the precipitate with cold dilute nitric acid appreciable quantities of silver were obtained, and the washing was continued till free from this metal. Warm nitric acid was found to render a further small quantity of silver soluble. The washing was continued till the reaction for silver was negative, and the precipitate was finally washed with dilute ammonia. The ammoniacal solution contained much dye, whilst the remaining precipitate was completely soluble. These results appear to indicate that compounds of silver other than silver chloride are present in the impure precipitate—compounds which raise the apparent quantity of chloride in flock. The precipitates of silvei chloride obtained from the extracts treated with perhydrol were quite white and pure.

The selected figures in Table III. show the trend of these results:

TABLE III.

Chlorine in Parts Per 100,000.

Sample.	Dyestuffs Present.	Dyestuffs Destroyed.	Difference.
1	12·9	10·4	24·0 per cent.
2	18·5	14·4	28·4 ,,
3	29·7	23·2	28·0 ,,

For Volhard's titration method, I find 500 to 250 c.c. of the extract a convenient quantity, according to the amount of chloride present. The concentration to 100 c.c. is conducted in a porcelain basin after the addition of a few drops of perhydrol and 1 c.c. of nitric acid, care being exercised to avoid local superheating. When cold, an excess of silver nitrate solution (strength, 1 c.c. = 2 mgrms. chlorine) is added and 5 c.c. of a saturated solution of iron alum, and then titrated with standard potassium thiocyanate solution equivalent in strength to the silver nitrate. The titration can be applied direct to the evaporated liquid, and the addition of nitric acid reduces or prevents the separation of flocculent matter.

The gravimetric method is tedious, and as the amount of the precipitate is frequently extremely small, the liability to error through loss of silver by reduction, etc., is considerable.

The direct titration with silver nitrate and potassium chromate is frequently inapplicable to evaporated extracts without filtration, which means an extra operation, whilst the end-point is less definite than that obtainable with Volhard's method. I therefore prefer to use Volhard's method of estimation, employing solutions of the strength mentioned, as, owing to want of definition of the end-point with weaker solutions, I was not able to get such close concordance between results obtained by the three methods. When the chlorine content is very small the direct titration serves as a useful safeguarding check, tending as it does to give results rather higher than the true figures.

All the foregoing experiments were conducted with distilled water, but it was found that, if suitable corrections were determined and applied, results in close agreement could be obtained by the use of ordinary water.

Variation in Chlorine Content of different Bales of a 1 Ton Consignment.—Through the courtesy of Messrs. R. and J. McCrae, Ltd., Glasgow, I was enabled to examine the contents of each of the forty bales of a 1 ton consignment. Each bale was carefully sampled, and an average sample examined. The results are as follows:

TABLE IV.

1 bale	contained	3	parts chlorine per 100,000.
3 bales	„	5	„
4 „	„	6	„
12 „	„	7	„
6 „	„	8	„
7 „	„	9	„
1 bale	„	10	„
3 bales	„	11 to 11·5	„
1 bale	„	13	„
1 „	„	17	„
1 „	„	27	„

The large variation between the chlorine content of the different bales is not surprising, as it is improbable that a large consignment is derived entirely from flocks subjected to the same washing.

The Regulations of the Rag Flock Act specify that a sample sent to an analyst must be "a well-mixed sample of flock." I have been informed that manufacturers or dealers in flock regard this regulation as a distinct grievance, and maintain that it should not be a "well-mixed sample of flock," but a "sample of well-mixed flock." Whatever disadvantage the present regulation inflicts on dealers, it is questionable whether the transposition of the words "well mixed" is desirable, as it would enable a manufacturer to mix bales of flock of low chlorine content with bales the chlorine content of which exceeded the standard, and then pass the whole consignment on the average chlorine figure, thereby defeating in a measure the purpose of the Act.

On the value of the chlorine standard as a reliable criterion of the comparative cleanliness of flock I express at the present time no opinion, but I claim that, in view of the present standard, the quality of a large consignment of flock can only be satisfactorily gauged by the sampling of each bale.

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Assay and Estimation of Aspirin. A. Astruc. (*J. Pharm. Chim.*, 1913, **8**, 5.)—A solution of 1·80 grms. of the sample in 50 to 60 c.c. of 90 per cent. alcohol, with 2 drops of alcoholic phenolphthaleïn, is titrated with $\frac{N}{1}$ potassium hydroxide: 10 c.c. (corresponding to 100 per cent. acetyl-salicylic acid) should be required. The mixture is then heated for not more than fifteen minutes on the water-bath, under a reflux condenser, with a further 20 c.c. of $\frac{N}{1}$ alkali, and the excess titrated back. The alkali used should again be 10 c.c. This saponification and second titration are necessary to detect substitution of the aspirin (acetyl-salicylic acid) by a mixture, for example, containing 76·66 per cent. of salicylic acid, with a neutral salt; such a mixture would give the same figure on direct titration as aspirin, but would, of course, not be saponified. O. E. M.

Estimation of Benzoic Acid in Meats. A. Krüger. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1913, **26**, 12-20.)—Since most of the methods proposed previously for the estimation of benzoic acid in foods fail to give satisfactory results in the case of substances containing a large proportion of protein, difficulty being experienced in extracting the whole of the acid from such foods, the author recommends the following method, having proved by experiment that it yields practically the whole of the benzoic acid actually present. Fifty grms. of the meat in a finely divided condition are mixed with 45 c.c. of 70 per cent. sulphuric acid and submitted to steam distillation; 500 c.c. of distillate are collected, the distillation flask being heated so as to maintain the volume of its contents as constant as possible during the distillation. The distillate is then filtered, rendered slightly alkaline with sodium hydroxide, and evaporated to a small volume. The residual liquid is heated on a water-bath, and potassium permanganate is added drop by drop until the pink coloration remains for five minutes. The excess of permanganate is destroyed by the addition of sodium sulphite, the mixture is evaporated to 10 c.c., transferred to a separating funnel, and acidified with sulphuric acid; the evaporating basin is rinsed with sodium sulphite solution and dilute sulphuric acid, the washings being added to the contents of the separating funnel. The acid solution, which should not measure more than 20 c.c., is extracted with ether, the ethereal extract is washed with water, the solvent allowed to evaporate spontaneously in a weighed basin, and the residue, after being dried for two hours over soda-lime, is weighed. The dried residue of benzoic acid may then be dissolved in alcohol, and the solution titrated with $\frac{N}{10}$ sodium hydroxide solution. When the weight of benzoic acid found is less than 30 mgrms., the sublimation method described by Polenske (ANALYST, 1911, **36**, 584) should be employed for the control estimation instead of the titration. W. P. S.

Investigations on Methods of Analysis of Cane Products. W. E. Cross. (*Louisiana Bulletin*, No. 135, p. 83.)—Both the Abbé and the immersion refractometer are recommended for determining the dry content of sugar-house products. Solid products are dissolved in an equal weight of water, some of the solution placed between the prisms of the Abbé instrument and the refractive index read. Syrups and molasses are tested direct, and molasses, if too dark to read even after treatment with "blankit," are diluted with an equal weight of fairly concentrated sugar solution (*cf.* Tischtschenko, *Zeitsch. Ver. Deuts. Zuckerind.*, **59**, 103). The maximum error of the method need not exceed ±0·1 per cent., or with high concentrations 0·05 per cent. The immersion refractometer is more limited in its application as it can only be used for concentrations up to 22 per cent.; for solutions of low concentration, however, and for standardising Brix spindles, its low maximum error recommends it. The use of dry lead subacetate for defecating sugar solutions is strongly recommended, not only on theoretical grounds, for it is admitted that substantially identical numbers are obtained by the use of a solution, but because of the simplification its use effects. For example, juices need only to be treated with the reagent, filtered, and polarised; the use of a lead solution involves two measurements, with attendant possibility of error. The use of dry lead acetate is also advantageous in determining sucrose in molasses by Clerget's method. In this method it is customary to use as little lead solution as possible, with the result that the polarisation is read on a dark solution with a considerable probability of error. Often an excess is employed of necessity without means being employed to eliminate the error due to the volume of the considerable precipitate produced. Deerr has proposed to reduce this error to negligible proportions by high dilution, but, unless a polarimeter which will accommodate a 600 mm. tube is available, the polarimeter reading is small, and any error in reading is multiplied. The authors add no unnecessary excess of lead, but continue the addition until maximum clarification is obtained. From the filtered solution, the excess of lead is removed by adding approximately the right amount of dehydrated oxalic acid. After filtration, the lead-free liquid is polarised before and after Clerget inversion. Andrlik's proposal to take the polarisation before inversion in acid solution, restricting the inversion of sucrose in the cold by addition of carbamide or betaine, has been tried but is not endorsed. The influence of betaine in restricting acid inversion at 28° C., the temperature of working in the tropics, is negligible, whilst that of carbamide, though marked, is far too weak to be of any service at 28° or even at 20° C. The Pellet-Lemeland method for the direct polarisation of sucrose was found to give results much below the truth, but a modification of it, consisting essentially in reducing the temperature of digestion from 100° to 55° C. is recommended for the routine analysis of low-grade products (*cf.* Cross and Taggart, ANALYST, 1912, **38**, 451). G. C. J.

Estimation of Crude Fibre and Cellulose. H. Matthes and F. König. (*Arch. der Pharm.*, 1913, **251**, 223-245.)—The authors have tested comparatively three methods for the estimation of crude fibre or cellulose, viz.: the Henneberg-Weende method, J. König's glycerol-sulphuric acid method, and Cross and Bevan's chlorination method. The material employed for the tests was powdered cinchona bark. By the Henneberg method there were obtained from the bark 27·85 per cent. of crude fibre, containing 50·17 per cent. of carbon and 6·48 per cent. of hydrogen; the ash in the product was 3·37 per cent. and protein 1·10 per cent. When extracted with boiling 50 per cent. alcohol the fibre lost 2·08 per cent., but on boiling with 94 per cent. alcohol there was a gain of 0·73 per cent., owing to the fixation of alcohol; the latter was removed by washing with ether and water. On digesting cinchona bark with glycerol-sulphuric acid according to König's method, there were obtained 29·26 and 28·47 per cent. of crude fibre and 16·04 and 15·68 per cent. of purified cellulose on further treatment with hydrogen peroxide. By Cross and Bevan's chlorination process, on the other hand, the bark yielded 21·49 per cent. of purified cellulose. The purified cellulose obtained by König's process contained 43·62 per cent. of carbon, that prepared by the chlorine process contained 45·29 per cent. of carbon. The investigations showed that the product obtained by König's process is not true cellulose, as claimed by the latter author, but a highly sensitive, modified derivative of cellulose. On repeating the treatments of the respective purified products, König's cellulose lost 20 per cent. in the second treatment with glycerol-sulphuric acid, whereas the chlorinated cellulose lost only 1·62 per cent. in a second chlorination. The cellulose obtained by chlorination is somewhat sensitive to alkalis and lost 4·38 per cent. on boiling with 1 per cent. sodium carbonate, but the chlorination process is far to be preferred to König's process for the estimation of cellulose, although the results of the latter may possess a certain "conventional" utility. That glycerol-sulphuric acid considerably attacks and dissolves cellulose was proved by experiments on filter paper. This lost 26·5 per cent. in the first treatment with the reagent, under the conditions prescribed by König, and a further 27·47 per cent. in a second treatment.

The authors give a detailed description of the chlorination method as applied by them to cinchona bark and recommended for the general treatment of vegetable materials. The substance is extracted with solvents, such as alcohol, water, and dilute sodium carbonate, and then dried *in vacuo*. About 0·2 to 0·3 grm. is placed in a beaker 12 to 15 cms. high and 6 to 7 cms. wide. Preferably a small spoon is weighed together with the material so that the latter may be spread out without loss. The beaker is covered with a moist filter paper and a watch-glass, and the contents are allowed to absorb moisture; after some time a little hot water may be added and worked into the powder with a glass rod until the material is thoroughly moistened, but not reduced to a thin paste. The mass is evenly distributed over the lower part of the beaker, then cooled in an ice-bath, and the beaker filled with chlorine gas. It is allowed to stand covered for twenty minutes in the ice-bath, and the excess of chlorine is blown out. The mass is again broken up with the rod, particular attention being paid to the darker portions, and the treatment with chlorine for twenty minutes is repeated a second and a third time, the entire duration of contact with the chlorine being one hour. The substance is then moistened with sulphurous acid, allowed to stand for one and a half hours, and collected and washed in a Gooch crucible. The product is then returned to the same beaker with 200 to 300 c.c. of a 2 per cent. solution of sodium sulphite with which it is digested on the water-bath for two hours. It is collected in a Gooch crucible, washed and treated in the crucible for five minutes with a 1 per cent. solution of permanganate; this is followed by sulphurous acid, the substance is washed, dried at 105° C., weighed and incinerated, the weight of the ash being deducted. J. F. B.

Volatile Constituents of Roasted Coffee. G. Bertrand and G. Weisweiller. (*Comptes rend.*, 1913, **157**, 212-213.)—Erdmann (*Ber.*, 1902, **35**, 1846) attributed the aroma of roasted coffee to the presence of traces of acetic acid, furfural, valeric acid, an unstable nitrogenous compound, and a phenolic body resembling creosote. The author, however, finds that these compounds by themselves are not sufficient to produce the aroma, but that pyridine is also present. The aqueous distillate from 5 kilos. of freshly roasted coffee was repeatedly distilled until the volatile constituents were concentrated to about 20 c.c. This was neutralised with hydrochloric acid, and the aqueous layer filtered and treated with barium silicotungstate. The precipitate, which was purified by two recrystallisations from water and dried at 30° C., was identified as pyridine silicotungstate. Various samples of commercial roasted coffee yielded from 0·2 to 0·25 grm. of pyridine per kilo., this proportion thus being greater than that of all other volatile compounds together. The essential oil separated from the aqueous part of the distillate required the addition of pyridine to reproduce the exact aroma of the coffee. C. A. M.

Estimation of Fat in Cheese. O. Allemann. (*Chem. Zeit.*, 1913, **37**, 876.)—A modified Soxhlet extraction method is recommended. The apparatus employed consists of a glass vessel which fits inside an ordinary Soxhlet apparatus; this vessel has a short, wide neck pierced with holes. Five grms. of the cheese and 20 c.c. of 25 per cent. hydrochloric acid are placed in the glass vessel and heated in a water bath until the cheese has dissolved. The vessel is then introduced into the Soxhlet apparatus, a small funnel is placed in the neck, the stem of the funnel being sufficiently long to reach to the bottom of the vessel, and the whole is attached to a reflux condenser. A weighed flask containing ether is connected to the lower end of the Soxhlet apparatus, and when the ether is heated to boiling, the condensed solvent from the condenser falls into the small funnel, passes down the stem, and rises drop by drop through the acid cheese solution. The ethereal solution collecting above the acid solution escapes through the holes in the neck of the vessel, when the latter is full, and passes back again into the flask in the usual way. The method yields concordant results, and the extraction of the fat is complete. W. P. S.

Analysis of Pastry containing Eggs. E. Nockmann. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1913, **25**, 717-726.)—The investigation recorded in this paper deals principally with the decomposition of lecithin which takes place in articles of food containing eggs. It is shown that the decrease in lecithin-phosphoric acid is due to the combined effect of heat and moisture during the preparation of such products as "noodles," etc., and is not brought about by heat or moisture alone, or by the time which the article is kept after it has been made. The quantities of total phosphoric acid and ether extract yielded by the products are not affected by the influence of heat and moisture. W. P. S.

Estimation of Essential Oils in Absinthe and Similar Liqueurs, and in Alcoholic Solutions of Essential Oils. A. Auguet. (*Ann. Falsif.*, 1913, **6**, 385-396.)—The French official method for the examination of liqueurs as regards their content of essential oil (essences) consists in distilling the sample, diluting the distillate to the original volume, and estimating its iodine absorption by Hübl's method. The author points out that the results obtained are much too low if the iodine solution employed is freshly prepared. It is known that Hübl's iodine solution gradually becomes acid on keeping owing to the formation of hydriodic acid, and the maximum results are obtained only when the solution is some months old. The addition of hydriodic acid to the iodine solution in the proportion of from 7·5 to 10 grms. per litre is, therefore, recommended. The following results were obtained with certain liqueurs, the figures expressing grms. of iodine absorbed per litre of the sample: absinthe, 2·9; anisette, 0·3 to 1·13; amer Picon, 0·15; peppermint, 0·39; menthe blanche, 0·47; bénédictine, 1·0. Alcoholic solutions containing 2 parts per litre of various essential oils gave results as follows: Balm-mint, 4·3; mint, 0·74; orange, 6·3; coriander (0·1 per cent.), 2·2; aniseed (French), 3·5; bergamot, 4·7; neroli (1·13 per cent.), 3·4. The time of contact of the iodine solution with the oil should be two hours at a temperature of about 18° C. Wijs' method of estimating the iodine absorption cannot be applied to alcoholic solutions of essential oils.

W. P. S.

Estimation of Lactose in Milk by Precipitation with Ammonium Sulphate. E. Kretschmer. (*Zeitsch. Physiol. Chem.*, 1913, **85**, 286-292.)—In the estimation of lactose according to Salkowski's method (ANALYST, 1912, **37**, 350), the application of the correction proposed by Jahnson-Blohm (*Zeitsch. Physiol. Chem.*, 1912, **83**, 441) is unnecessary. Removal of proteins by Weyls' process (*Zeitsch. Physiol. Chem.*, 1903, **65**, 246), or by Oppenheim's method (ANALYST, 1909, **34**, 500), also gives good results, whilst Allihn's gravimetric method and Hoppe-Seyler's lead acetate method give lower results than Salkowski's method.

E. W.

Aluminium Hydroxide as a Protein Precipitating Reagent in the Estimation of Lactose in Milk. W. H. Welker and H. L. Marsh. (*J. Amer. Chem. Soc.*, 1913, **35**, 823-824.)—A thin jelly of aluminium hydroxide ("alumina cream") may be used generally for the precipitation of colloids from their solution, and may be employed for the removal of proteins from milk in the estimation of lactose in the latter. The weighed portion of the milk is treated directly with the aluminium hydroxide, shaken, diluted to a definite volume, and filtered, the lactose being then estimated in the filtrate by means of Fehling's solution. The use of an excess of aluminium hydroxide does not affect the results, and the filtration is more rapid than when the proteins are precipitated with copper sulphate and alkali.

W. P. S.

Composition of Different Varieties of Red Pepper. L. M. Tolman and L. C. Mitchell. (*U.S. Dept. of Agric., Bureau of Chem., Bull.* No. 163, May, 1913, pp. 1-32.)—Analyses of African and Japanese cayenne pepper or chillies, Hungarian

paprika, and Spanish pimenton or pimiento, are recorded. A summary of the results obtained is given in the following tables :

CAYENNE PEPPER OR CHILLIES.

	African Chillies.		Japanese Chillies.	
	Minimum per Cent.	Maximum per Cent.	Minimum per Cent.	Maximum per Cent.
Total ash	4·86	6·16	5·06	5·82
Ash insoluble in HCl	0·37	0·85	0·27	0·53
Non-volatile ether extract	16·07	19·68	17·67	22·89
Crude fibre	25·39	29·46	22·86	26·08

HUNGARIAN PAPRIKA (A VARIETY OF *Capsicum annuum L.*).

	Shells, Seeds, Placentæ, and Stems.		Shells, Seeds, and Placentæ.		Seeds and Placentæ		Shells.		Stems.	
	Min. per Cent.	Max. per Cent.	Min. per Cent.	Max. per Cent.	Min. per Cent.	Max. per Cent.	Min. per Cent.	Max. per Cent.	Min. per Cent.	Max. per Cent.
Loss at 70° C. *in vacuo*	3·29	3·76	3·11	4·16	3·73	4·23	3·44	4·00	4·66	5·78
Total ash	5·08	6·03	4·66	5·56	3·38	3·80	6·29	7·11	9·84	11·35
Ash insoluble in HCl	0·24	0·33	0·20	0·31	0·21	0·30	0·22	0·37	0·66	1·20
Non-volatile ether extract	12·21	16·43	13·94	17·35	25·97	27·56	5·14	6·90	2·11	3·05
Crude fibre	20·69	22·76	20·47	23·18	20·89	21·60	22·20	24·07	29·34	32·86

SPANISH PIMENTON (ALSO A VARIETY OF *Capsicum annuum L.*).

	Shells, Seeds, Placentæ, and Stems.		Shells, Seeds, and Placentæ.		Shells.		Seeds and Placentæ.		Stems.	
	Min. per Cent.	Max. per Cent.	Min. per Cent.	Max. per Cent.	Min. per Cent.	Max. per Cent.	Min. per Cent.	Max. per Cent.	Min. per Cent.	Max. per Cent.
Loss at 70° C. *in vacuo* ...	4·31	5·98	4·52	5·09	4·74	5·02	3·59	4·12	4·86	5·98
Total ash	6·98	7·86	6·60	7·35	7·55	8·46	4·43	5·02	13·00	15·77
Ash insoluble in HCl ...	0·29	0·48	0·24	0·40	0·26	0·37	0·22	0·31	0·58	1·07
Non-volatile ether extract ...	11·30	12·58	11·58	13·34	5·44	6·81	21·82	24·58	1·22	2·05
Crude fibre	19·53	20·59	18·76	20·34	17·26	18·70	19·90	24·08	27·66	30·98

W. P. S.

Phytic Acid in Cottonseed Meal and Wheat Bran. J. B. Rather. (*J. Amer. Chem. Soc.*, 1913, **35**, 890-895.)—The author has investigated the phosphorus compounds of wheat bran and cottonseed meal which are soluble in 0·2 per cent. hydrochloric acid and the phosphorus compounds of cottonseed meal soluble in

0·2 per cent. ammonia after extraction with hydrochloric acid. The method of purifying the phosphorus compounds was, briefly, as follows: The compound was precipitated in acid solution with copper acetate, the copper was removed as sulphide, and the solution evaporated. The residue was dissolved in a small quantity of water, treated with ten times its volume of ammonia, and the mixture allowed to stand over-night. After filtration, the ammonia was removed from the filtrate by evaporation, and the aqueous solution of the residue was precipitated a number of times with barium chloride in the presence of alkali. The free acid was, finally, recovered from its copper salt, and obtained as a syrup on the evaporation of its alcoholic solution. Analyses were made of the products obtained from the two sources mentioned, and from the results obtained the authors propose the formula $C_{12}H_{41}O_{42}P_9$ for the inositol-phosphoric acid, or so-called phytic acid, of feeding stuffs. The properties of the silver salts prove that the inositol-phosphoric acids of wheat bran and cotton-seed meal are identical. There is no evidence that wheat bran contains an inositol-phosphoric acid with pentose in the molecule. W. P. S.

Saffron containing Formaldehyde. J. Bulir. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1913, **26**, 43-44.)—Certain samples of saffron examined by the author were found to contain formaldehyde; these samples were at the same time adulterated with from 25 to 30 per cent. of glycerol, and the formaldehyde was apparently added as a preservative. Other samples contained from 23 to 40 per cent. of added invert sugar, whilst a few samples were adulterated with sucrose, the amount of the latter varying from 10 to 18 per cent. Samples yielding normal amounts of ash, in spite of the fact that they were adulterated with sugars, were found to contain, in addition to the latter, quantities of borax and sulphates. The ash of genuine saffron yields only a trace of boric acid and about 10 per cent. of sulphuric acid (SO_3), but the ash of the samples in question contained from 25 to 30 per cent. of sulphuric acid.

W. P. S.

Detection and Estimation of Salicylic Acid in Fruit Juices. W. Heintz and R. Limprich. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1913, **25**, 706-717.)—Having found that the method described by Vierhout (ANALYST, 1911, **36**, 411) is untrustworthy, the authors propose a method which depends on the fact that, under given conditions, a definite and constant proportion of the amount of salicylic acid present in a dilute alcoholic solution may be extracted with petroleum spirit, and that, on shaking the petroleum spirit solution with ferric chloride solution, the whole of the acid passes into the aqueous layer in the form of its violet-coloured iron compound. The details of procedure are as follows: Twenty-five grms., or more, of the fruit juice are placed in a 250 c.c. stoppered cylinder, diluted to 50 c.c. with water, acidified with a few drops of sulphuric acid, and shaken with 100 c.c. of petroleum spirit. Fifty c.c. of 96 per cent. alcohol are then added, the mixture is again shaken, and allowed to separate into layers. Ten c.c. of the petroleum spirit layer are then transferred to a tube and mixed with 10 c.c. of a freshly prepared 0·1 per cent. ferric chloride solution, the coloration obtained being compared with that given by known quantities of salicylic acid. These comparison solutions are prepared by shaking 50 c.c. of a

0·1 per cent. aqueous salicylic acid solution with the quantities of sulphuric acid, petroleum spirit, and alcohol mentioned, and, after separation has taken place, portions of 0·5, 1·0, 1·5, 2·0, c.c., etc., of the petroleum spirit layer are transferred to separate tubes, and each mixed with 10 c.c. of the ferric chloride solution. The violet coloration obtained retains its intensity for at least twenty-four hours. The method may also be applied to the estimation of salicylic acid in jams, beer, wine, etc., but in the case of samples containing alcohol, this must be removed by evaporation in the presence of alkali before the estimation is commenced (*cf.* ANALYST, 1905, **30**, 124). W. P. S.

Physico-Chemical Estimation of Sulphates, Tartaric Acid, Calcium, and Magnesium in Wine. Duboux. (*Chem. Zeit.*, 1913, **37**, 879.)—Sulphates may be estimated accurately in wine by the electrical conductivity method (ANALYST, 1909, **34**, 56) when barium chloride solution is employed as the precipitant. For the estimation of tartaric acid, the wine should be treated with alcohol and acetic acid, and then titrated with barium acetate solution. The separation of tartaric, malic, and succinic acids is attained by precipitating the three acids together from the wine by means of silver nitrate. The precipitate is dissolved in sodium bromide solution, and the solution of the sodium salts thus obtained is treated as follows: The total acids are estimated in an aliquot portion of the solution with $\frac{N}{2}$ lanthanum nitrate solution after the addition of alcohol. In another portion the tartaric acid and malic acid are estimated in acetic acid solution with the same reagent, and the tartaric acid alone is titrated in strong acetic acid and alcoholic solution with barium acetate solution, the conductivity curves being plotted in each case. For the separation of calcium and magnesium, 50 c.c. of the wine are mixed with 100 c.c. of alcohol and a few drops of sulphuric acid; the precipitated calcium sulphate is collected, dissolved in water, and titrated with potassium oxalate solution. The filtrate is treated with lead nitrate to remove sulphates and phosphates, evaporated, and the residue ignited. The ash is then dissolved in water in the presence of ammonia and ammonium chloride, and the solution is titrated with phosphoric acid. W. P. S.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Micro-Calorimeter for Measuring Heat Evolved by Bacteria. K. v. Körösy. (*Zeitsch. Physiol. Chem.*, 1913, **86**, 383-401.)—Advantage is taken of the latent heat of vaporisation of a suitable liquid, and the amount of heat evolved estimated by measuring the volume of liquid evaporated. The calorimeter consists essentially of a double-walled vessel, the annular space of which communicates with a graduated glass tube, and after being evacuated is filled with pure ether. The whole is immersed in a water-bath maintained at constant temperature, the double-walled vessel being protected by a large glass envelope, and thus thermally isolated by a layer of air whilst the graduated glass tube is in direct contact with the water. When heat is generated in the calorimeter ether distils over into the graduated tube. The apparatus can be used over a fair range of temperatures. E. W.

New Reagent for the Detection of Traces of Blood. R. F. Ruttan and R. H. M. Hardisty. (*Canadian Med. Assoc. J.*, September, 1912; through *Chem. Zentralbl.*, 1913, I., 2182-2183.)—A 4 per cent. solution of *o*-toluidine in acetic acid was found to be a very sensitive reagent for blood in the presence of hydrogen peroxide. One part of blood in 7,000,000 parts of solution can be detected by the aid of this reagent, whilst the guaiacum test will detect 1 part in 50,000, and the benzidine test 1 part in 700,000. The *o*-toluidine reagent is also the most sensitive test for the detection of blood in urine, stomach-contents, etc., being capable of detecting 1 part in 24,000, whilst the other two reagents mentioned will not give a reaction with less than 1 part of blood in 6,000 parts of fluid. W. P. S.

Method of Standardising Disinfectants with and without Organic Matter. J. F. Anderson and T. B. McClintic. (*U.S. Treasury Dept., Hygienic Laboratory Bulletin* No. 82, 1-34.)—The method described and recommended is a compromise between the Rideal-Walker method and the "Lancet" method, by which is meant the method worked out by Sims Woodhead and his collaborators (*Lancet*, 1909, **177**, 1454, 1516, 1612; ANALYST, 1910, **35**, 405). The most important deviation from the Rideal-Walker method is in the method of calculating the coefficient. Here the authors follow the "Lancet" method in defining the coefficient as the mean of two ratios—one the result of experiments in which the organism is exposed to the diluted disinfectants for two and a half minutes, the other the result of experiments where the exposure is much longer; but they make their longest period only fifteen minutes, as against the thirty minutes of the "Lancet" method. The most important deviation from the "Lancet" method is in the rejection of *B. coli* as standard organism, together with bile-salt media, and the return to *B. typhosus* and extract broth, as in the Rideal-Walker method. It is held, on the one hand, that various strains of *B. typhosus* differ less in resistance to disinfectants than do various strains of *B. coli communis*; and, on the other, that the unmodified Rideal-Walker method is unsatisfactory, since the time-period at which the comparison is to be made is not defined, so that an operator can, on the same day and with the same materials, make the coefficient of some one disinfectant anything from 9 to 12.

The following are the more important particulars of the method described: The medium used is extract broth of reaction +1·5; the test organism *B. typhosus* (Hopkins), the result of subculturing in extract broth on at least three successive days, using a 4 mm. loopful for carrying the culture over. The temperature of medication is precisely 20° C., and is controlled by the use of a water-bath. To 5 c.c. of each dilution of the disinfectant and of the phenol controls precisely 0·1 c.c. of the filtered culture is added by means of a pipette. Experiments are described which show that the unmeasured "drop" of the Rideal-Walker method may introduce large errors, whilst the disadvantages of the "Lancet" "spoons" are pointed out. For the inoculation of the subcultures a 4 mm. loop of 23 (U.S.) gauge wire is used. The subcultures are incubated forty-eight hours at 37° C. The actual dilutions of the disinfectant to be employed necessarily vary with its strength, but they are to progress as follows: By a difference of 5 up to 1:70, by 10 from 1:70 to 1:160, by 20 to 200, by 25 to 400, by 50 to 900, by 100 to 1,800, by 200 to 3,200. Subcultures are made every two and a half minutes up to fifteen minutes. To determine the coefficient, the figure representing the degree of dilution of the weakest strength of the disinfectant that kills within two and a half minutes is divided by the figure representing the degree of dilution of the weakest strength of the phenol control that kills within the same time. The same is done for the weakest strength that kills in fifteen minutes. The mean of the two is the coefficient.

Disinfectants with a phenol coefficient exceeding unity should also have their coefficient determined in presence of organic matter. As types of organic matter the authors use peptone (Witte) and gelatine (Best French Gold Label). They describe a convenient way of making the addition of organic matter without complicating the calculation of the dilution of the disinfectant or introducing unnecessary operations. A stock solution containing 10 per cent. peptone and 5 per cent. gelatine is prepared. The disinfectant dilutions are all made 25 per cent. stronger than they are required to be finally, and 4 c.c. portions instead of the usual 5 c.c. portions are transferred to the medication tubes in the water-bath at 20 c.c. A convenient quantity (15 c.c.) of the peptone-gelatine medium is transferred to a test-tube and also placed in the bath at 20° C., and to it one-tenth of its bulk of the culture of *B. typhosus* is added. The medication tubes are then each seeded with 1 c.c. of this mixture. The dilution is now that required, and the percentage of organic matter in each tube identical—namely, 3 per cent. G. C. J.

Precipitation of Enzymes from their Solutions by Means of Moist Aluminium Hydroxide. W. H. Welker and J. Marshall. (*J. Amer. Chem. Soc.*, 1913, **35**, 822.)—Aluminium hydroxide in the form of a thin jelly completely precipitates enzymes from their solutions. In the experiments carried out by the authors equal volumes of the enzyme solution and the reagent were shaken together for a few seconds and then filtered. In only one case—namely, amylase (from saliva)—was the precipitation incomplete, but peroxydase, oxydase, pepsin, rennin, trypsin, and lipase were removed completely from their solutions. The only zymogen studied was pepsinogen, and this was precipitated with the greatest difficulty from its solution in glycerol. W. P. S.

Microscopical Detection of Protoplasmalipoids, especially in Muscular Tissue. A. Noll. (*Arch. f. Anat. u. Phys.* (*Waldeyer-Rubner*), *Physiol. Abt.*, 1913, 35-55; through *Chem. Zentralbl.*, 1913, II., 381-382.)—The author accomplishes the histological separation of the protoplasmic fat from its protein envelope by digestion of the latter in a 0·1 per cent. solution of pepsin containing 0·3 per cent. of hydrochloric acid, or by solution of the protein in 15 per cent. ammonium chloride or 5 per cent. magnesium sulphate solution. After twenty-four hours' treatment by either method, microscopical fat globules, which give the osmic acid and other characteristic reactions, may be readily observed, whether the original material be skeletal muscle of man, mammals, birds, amphibia or insects, or cardiac tissue or unstriated muscle. G. C. J.

Estimation of Absorbed Bases in Soils. D. Prianischnikow. (*Landw. Versuch-Stat.*, 1913, 79-80, 667-680; through *Chem. Zentralbl.*, 1913, I., 2173-2174.)—In Kellner's method for the estimation of absorbed bases in soils (displacement with ammonium chloride) the ammonium chloride may be replaced by ammonium nitrite which, although having the disadvantage of being unstable, displaces more potash from the soil than does the chloride; ammonium acetate and ammonia (3·2 per cent. solution) may also be employed. The absorbed ammonia in soils may be estimated by extraction with a 5 per cent. potassium chloride solution. Further experiments with different minerals showed that nepheline yields the largest quantity of potash (K_2O), then follow biotite, muscovite, orthoclase, sanidine, microline, and leucite, the latter giving the least quantity. These minerals may be classed in a somewhat similar order as regards their availability as plant foods. Artificial zeolites were found to be almost useless as a plant food when used alone, but after the addition of other nutritive salts the potash contained in the zeolites was assimilated readily.

W. P. S.

Precipitation of Uric Acid and Purin Bases by Zinc Salts. E. Salkowski. (*Zeitsch. Physiol. Chem.*, 1913, **85**, 346-347.)—In addition to uric acid the purin bases in urine are almost completely precipitated by zinc salts. E. W.

ORGANIC ANALYSIS.

Purification of Acetone by Means of Sodium Iodide. K. Shipsey and E. A. Werner. (*J. Chem. Soc.*, 1913, **103**, 1255-1257.) — About 440 grms. of boiling acetone are required to dissolve 100 grms. of anhydrous sodium iodide. On cooling to room temperature, crystals, having the composition $NaI.3C_3H_6O$, separate, and on cooling to $-8°$ C., a larger crop of crystals (nearly 200 grms.), containing a still larger proportion of acetone of crystallisation, may be obtained. From these crystals, after separation from the mother liquor, nearly 120 grms. of pure acetone may be obtained by simple distillation. If the original "acetone" has a sp. gr. at 0°/4° C. of 0·815, a boiling-point range of from 55·9 to 56·3° C., and a strong reducing reaction on permanganate, the acetone recovered by this process, after drying over calcium chloride and redistillation, will boil at 56·1-56·2° C., possess a sp. gr. of 0·8130, and will withstand the permanganate test for fifteen minutes. The sodium iodide can be used repeatedly. After being used once with "acetone" not quite dry, it will contain more or less of the dihydrate, and will then dissolve in less than 4·4 times its weight of boiling acetone, the dihydrate being soluble in less than its own weight. It is not advisable to make stronger solutions than 50 per cent., even when the dihydrate stage has been reached, as it is then difficult to get all the impurities into the small bulk of mother liquor. From 100 grms. of the dihydrate and 100 grms. of acetone, about 140 grms. crystals may be obtained, and from these crystals 70 grms. of pure acetone recovered. G. C. J.

Application of Young's Method for the Preparation of Absolute Alcohol. G. Chavanne. (*Bull. Soc. Chim. Belg.*, 1913, **27**, 205-209.)—The two isomeric acetylene dichlorides form binary mixtures with absolute alcohol, and ternary mixtures with water and alcohol, boiling at low temperatures. They can thus be used instead of benzene in Young's method of obtaining absolute alcohol. For example, 500 grms. of 92·4 per cent. alcohol were distilled with 1,750 grms. of a mixture of the dichlorides (containing about 75 per cent. of the less volatile chloride of b.-pt. 60·25° C.) which had previously been dried over calcium chloride. A Young's dephlegmator with eight sections was used, and the distillation done at the speed of 1 drop per second. The yield of 99·1 per cent. alcohol was 300 grms. The percentage compositions of the binary and ternary mixtures with the two compounds are as follows:

	Isomer of B.-Pt., 48·35° C.				Isomer of B.-Pt., 60·25° C.			
	Boiling-Point.	Composition.			Boiling-Point.	Composition.		
		$C_2H_2Cl_2$.	Alcohol.	Water.	° C.	$C_2H_2Cl_2$.	Alcohol.	Water.
Ternary mixture	44·4	94·5	4·4	1·1	53·8	90·5	6·65	2·85
Binary mixture ...	46·5	94·0	6·0		57·7	90·2	9·80	
Binary mixture (chloride and water)	45·3	98·1		1·9	55·3			3·35

C. A. M.

Method for Estimating Water in Alcohol. Equilibria in Systems containing Alcohols, Water, and Potassium Fluoride. G. B. Frankforter and F. C. Frary. (*J. Phys. Chem.*, 1913, **17**, 402-473; through *Chem. Zentralbl.*, 1913, II., 421-423.)—Potassium fluoride is capable of salting out ethyl alcohol from its aqueous solutions. This is explained by an examination of the diagrams showing the relationships between methyl, ethyl, and propyl alcohol towards water and potassium fluoride and carbonate at the ordinary temperature. The solubility of potassium fluoride in absolute alcohol decreases somewhat on the addition of water, and then increases again. From the solubility curves of potassium fluoride in aqueous alcohol, the following method of estimation has been deduced: A glass-stoppered flask is charged with a weighed quantity of freshly ignited potassium fluoride and a known quantity of the aqueous alcohol to be examined. The contents of the flask are shaken until the fluoride has dissolved and the liquid has separated into two layers. Then water is added whilst shaking, until the two liquids again form a homogeneous mixture; the quantity of water added is then ascertained by weighing. From these data, by the aid of a table, the proportion of water in the aqueous alcohol may be estimated with a fair degree of accuracy. A small quantity of methyl alcohol can be allowed for in the calculation. Potassium fluoride is a better drying agent for liquids than the carbonate, because the vapour tension of its saturated solution is lower than that of a saturated solution of potassium carbonate. Anhydrous potassium fluoride is also to be preferred to the carbonate as a dehydrating agent on account of its greater solubility in organic liquids. J. F. B.

Microsublimation of Alkaloids under Reduced Pressure. R. Eder. (*Schweiz. Wochensch. f. Chem. u. Pharm.* 1913, **51**, 228, 241, 253; through *Chem. Zentralbl.*, 1913, II., 91-92.)—The temperature at which alkaloids sublime under reduced pressure and the appearance of the sublimates are valuable aids to their identification. The material extracted and purified by the aid of various solvents in the usual manner is sublimed under a pressure of 10 mm. in one or other of two pieces of apparatus which are described in the paper, and which allow the temperature of sublimation to be noted as well as the appearance of the sublimate as it accumulates. The following temperatures of sublimation are given: Cocaine 75-90° C., atropine 93-110° C., codeine 100-130° C., quinine 133-148° C., narcotine 146-156° C., brucine 158-175° C., solanine 168-184° C. The apparatus is partly immersed in a sulphuric acid bath, the temperature of which is slowly raised to a point about 20-25° C., above that at which a sublimate is first noted, but in no case above the melting-point of the substance. The sublimate is meanwhile observed through a lens. According to their behaviour under this sublimation test, alkaloids can be divided into five classes. To the first class belong those which, without melting, give rise to a sublimate which is immediately recognisable as crystalline. Caffeine, theobromine, cinchonine, solanine, and cantharidine fall into this class. Those which, without melting, give rise to a sublimate which at first appears to be amorphous, but in which crystals can be recognised before the end of the experiment, form a second class which includes strychnine, morphine, apomorphine hydrocloride, codeine, thebaine, narcotine, pilocarpine hydrocloride, yohimbine, cinchonidine, quinidine, quinine, conine hydrobromide, arecoline hydrobromide, and hyoscyamine. The alkaloids of the third class which, without melting, give rise to an amorphous sublimate which rarely develops crystals, are divided into two subclasses, III*a* and III*b*. The alkaloids of class III*a*—cocaine, brucine, papaverine, piperine, atropine, homatropine, physostigmine, and hydrastine—may develop a few crystals, whereas those of class III*b*—scopolamine, hydrobromide, aconitine, and delphinine—always remain amorphous. The fourth class consists of those which only sublime above their melting-point, and includes narceine, pilocarpine, veratrine, emetine, and colchicine; whilst the fifth class is formed of those which do not sublime, the only examples given being nicotine hydrochloride and sparteine sulphate. G. C. J.

Precipitation of Alkaloids by Lloyd's Reagent. S. Waldbott. (*J. Amer. Chem. Soc.*, 1913, **35**, 837-838.)—The reagent is essentially hydrated aluminium silicate, derived from fullers' earth; it possesses the property of precipitating alkaloids completely from their neutral or acid solutions. The alkaloid may be recovered by treatment with an alkali and an alkaloid solvent. The activity of the reagent is not impaired by the action of concentrated nitric acid or *aqua regia*, but it is rendered inert when heated to redness. The reagent is also capable of precipitating inorganic salts, such as barium chloride, lead acetate, zinc sulphate, etc. In the course of the investigation it was found that quinine sulphate is precipitated when its solution is mixed with colloidal silicic acid or with colloidal arsenious sulphide. W. P. S.

New Indicator. F. Aron. (*Pharm. Post*, 1913, **46**, 521-522; through *Chem. Zentralbl.*, 1913, II., 381.)—Orthodihydroxydibenzalacetone—$OH.C_6H_4.CH : CH.CO.CH : CH.C_6H_4.OH$—is pale yellow in acid solution, orange red in alkaline solution. The transition is sharp. Three or four drops of a 1 per cent. alcoholic solution to 100 c.c. of liquid to be titrated is a suitable quantity. The indicator cannot be used in presence of ammonia. G. C. J.

Estimation of Iodine in Organic Substances. F. Blum and R. Grützner. (*Zeitsch. Physiol. Chem.*, 1913, **85**, 429-471.)—The detection and estimation of minute quantities of iodine in the presence of large amounts of organic matter, a condition in which it may occur in blood, present difficulties, and the usual methods of halogen estimation fail. Baumann's method of incinerating by fusion with a mixture of soda and saltpetre is effective, and is serviceable if only small quantities of organic substance are used, as in urine or thyroid glands. With larger quantities the method becomes impracticable, owing to the amount of alkali salts introduced. In order to avoid this the author recommends incinerating with barium peroxide. The substance is moistened with small quantities of pure sodium hydroxide and sodium carbonate, and dried in the iron crucible used for ignition. It is then mixed well with barium peroxide—in the case of blood 50 grms. per 100 c.c.—and the mixture gradually introduced into one or several iron crucibles. The reaction must not be allowed to become too violent, and when no more smoke issues fresh peroxide is added until further addition is not accompanied by flame. The excess of peroxide is destroyed by adding pure animal charcoal in excess. The heating should not be continued too long, and all operations must be carried out in a covered crucible. The contents of the crucible after cooling are transferred by means of hot water into a beaker, and carbon dioxide passed through until the barium is completely precipitated. The removal of the barium can be hastened by adding sodium sulphate. After complete precipitation the mass is filtered, and washed with hot water containing some sodium carbonate or sulphate. The filtrate is treated with excess of potassium permanganate, and after addition of a little talc, boiled for ten to thirty minutes, according to the amount of reducing substance present. After complete alkaline oxidation sulphuric acid (1 : 4) is added until distinctly acid, and boiling continued for five to fifteen minutes. After cooling the liquid is made alkaline with soda and alcohol added to completely reduce excess of permanganate. The mass is then boiled until the odour of alcohol disappears, filtered, the filtrate boiled for five minutes to remove last traces of alcohol, made acid with phosphoric acid, and again boiled, usually with addition of ammonium sulphate, to insure complete decomposition of nitrites, although in most cases this is not necessary. This solution contains the iodine in the form of iodic acid, and is free from other oxidising substances. It is made acid to congo-paper with sulphuric acid, potassium iodide added, and the liberated iodine titrated with thiosulphate. The iodine liberation should be instantaneous; a slow liberation indicates traces of nitrous or possibly hypochlorous acids. It is advisable to test a portion of the solution, and if necessary to boil with ammonium sulphate before titration. The author found that even the purest phosphoric acid contained some reducing impurity; this can be rendered harmless by boiling with $\frac{N}{10}$ permanganate, and decomposing the excess of permanganate with a few drops of $\frac{N}{10}$ oxalic acid. In order to distinguish between inorganic and organic iodine (iodine contained in proteins) in blood, the blood is mixed with 4 volumes of acetone. This completely precipitates the proteins, whilst the inorganic iodine remains in the acetone solution. After standing for one hour the coagulum is filtered off, and washed with 80 per cent. acetone. The acetone solution is made strongly alkaline by adding a few c.c. of 30 per cent. sodium hydroxide, distilled, and the residue treated as described above. E. W.

Extract of Male Fern. C. A. Hill. (*Paper read before Brit. Pharm. Conf.*, 1913; through *Pharm. J.*, 1913, **91**, 126-128.)—The following results are typical of those obtained in the analysis of twenty genuine samples of extract of male fern, and of three adulterated samples:

	Sp. Gr. at 15° C.	Refractive Index at 40° C.	Loss on Drying at 100° C.	Insoluble in 10 Vols. of Petroleum Spirit.	Crude Filicic Acid.	"Potash Extract."	"Potash Insoluble."
			Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.
enuine.. ...	1·0235	1·5006	2·69	7·0	25·27	39·5	57·81
,, ...	1·009	1·4965	3·65	7·5	22·0	42·95	53·4
,, ...	0·9985	1·4960	4·64	2·0	24·5	48·5	46·86
dulterated ...	0·998	1·4869	—	74·0	13·2	—	—
,, ...	0·9829	1·4823	5·03	About 60	11·6	21·43	73·54
,, ...	1·006	1·4874	2·57	65·0	14·1	27·13	70·3

As a rule the sp. gr. is higher than 1, and this value is lowered by the addition of chlorophyll to improve the colour of an extract. The refractive index should not be less than 1·49, while the loss on heating should not exceed 6 per cent. The portion insoluble in petroleum spirit (which should be separated in a centrifuge) should not exceed 20 per cent. Crude filicic acid or filicin, when estimated by the baryta method of the Swiss Pharmacopœia, averages about 22 per cent., and should not be less than 20 per cent. In estimating the "potash extract" and "potash insoluble," about 20 grms. of the sample are dissolved in ether and repeatedly shaken with 1 per cent. potassium hydroxide solution until nothing more is extracted. The united alkaline extracts are washed with ether, and the washings added to the original ethereal solution, which is then evaporated. The residue (potash insoluble) is dried and weighed. The alkaline liquid is acidified with hydrochloric acid and extracted with ether, the extract evaporated, and the residue (potash extract) dried and weighed. The "potash extract" includes the filicic acid, and by extracting it with 3 per cent. baryta solution, the same result will be obtained as by estimating the filicic acid by the Swiss Pharmacopœia method. In the "potash insoluble" the bulk of any fixed oil used as an adulterant will be found. In the case of genuine male fern extract this portion consists almost entirely of triglycerides. The following values were obtained with the extracts from three genuine samples and one adulterated with 59 per cent. of castor oil:

Specific Gravity.	Refractive Index at 40° C.	Saponification Value.	Saponification Value after Acetylation.	Acetyl Value.	Hydroxyl Value.	Iodine Value.
0·9387	1·4715	173·7	198·8	28·6	29·2	—
0·9340	1·4729	175·3	207·2	36·1	37·1	120·6
0·9372	1·4727	177·4	204·9	31·7	32·5	121·2
0·9555	1·4732	170·7	281·1	127·2	140·5	—

C. A. M.

Analytical Constants of Extract of Male Fern. E. F. Harrison and P. A. W. Self. (*Paper read before Brit. Pharm. Conf.*, 1913; through *Pharm. J.*, 1913, **91**, 128-129.)—Ten specimens of extract prepared in the laboratory from commercial samples of material gave values falling within the following limits: sp. gr., 1·018 to 1·052; refractive index at 20° C., 1·4995 to 1·5157; saponification value, 227 to 259; insoluble in 10 vols. of petroleum spirit, 4·1 to 6·7 per cent.; unsaponifiable matter, 4·1 to 6·7 per cent.; and crude filicin (by Swiss Pharmacopœia method), 19·3 to 28·0 per cent. None of these genuine samples would thus comply in every particular with the limits prescribed by Parry (*Pharm. J.*, 1911, **89**, 778). Estimation of the combining weight of the liberated fatty acids, as recommended by Parry, was found unsatisfactory, since it was not practicable to wash the whole of the mineral acid from the ethereal solution without removing also part of the fatty acids. C. A. M.

Hydrolysis of Lævulosans and its Application for Analytical Purposes. P. L. de Vilmorin and F. Levallois. (*Bull. Soc. Chim.*, 1913, **13**, 684-691.)—The authors have investigated quantitatively the saccharification by acids of lævulosans, employing purified inulin as a typical representative of the group. The use of sulphuric acid and oxalic acid at various concentrations and at temperatures ranging from 75°-100° C. gave results too variable for analytical use, although it was evident that the hydrolysis tends to reach a limit with the production of 89 to 90 per cent. of reducing sugar calculated on the weight of the inulin. The reaction is complicated by the destruction of the lævulose at the high temperature in presence of the acid. Perfectly concordant results are obtained with acetic acid at concentrations between 3 and 10 per cent. at a temperature of about 80° C., and numbers about 1 per cent. higher are obtained at 100° C. The action of acetic acid is, however, very slow, requiring eleven hours or more to reach the maximum limit; moreover, the use of such large proportions of acid introduces difficulties when the liquid is neutralised for the estimation of reducing sugars. By far the most satisfactory inverting agent is sulphosalicylic acid, at concentrations ranging from 0·72 to 4·3 grms. per litre and at temperatures between 80° and 100° C. The limit of hydrolysis is reached at 80° C. in seventy-five to ninety minutes, and the results remain constant when heating is prolonged for eight or nine hours. At 97° C., with acid at $\frac{1}{100}$ grm. mol. concentration, the limit is reached in fifteen minutes and remains unchanged for two hours. Sulphosalicylic acid, moreover, presents no difficulties in the subsequent estimation of the sugar by Fehling's solution.

J. F. B.

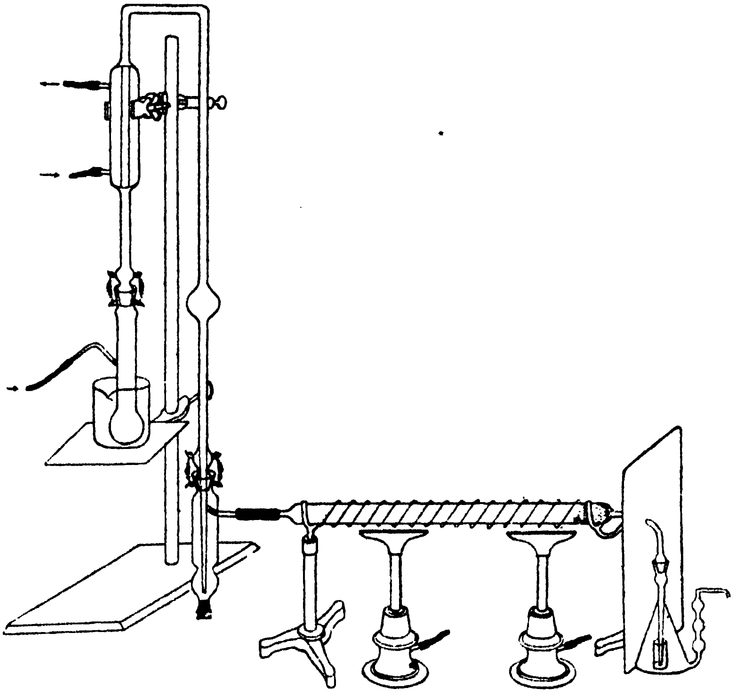
Preparation and Analysis of Methane. C. Campbell and A. Parker.

(*J. Chem. Soc.*, 1913, **103**, 1292.)—To estimate the hydrogen in methane prepared from aluminium carbide, a small quantity of oxygen was added, and the contraction observed on passing the gases over palladium black at 90° to 100° C. The apparatus devised by Bone and Wheeler was used. The palladium was contained in a small bulb, heated in a water-bath, and connected on one side to a small laboratory vessel, and on the other, through a bulb to intercept mercury, to the measuring vessel. A three-way tap allowed "connection" between the measuring vessel and the main laboratory vessel on the palladium bulb. The palladium bulb was first exhausted by connecting it three or four times with the exhausted measuring vessel, which has about twenty times its capacity, after all gas had been removed from the rest of the apparatus. The methane was then measured, mixed with a small quantity of oxygen, and passed slowly two or three times over the palladium; it was then pumped back into the main laboratory vessel, passed back into the apparatus, and measured.

O. E. M.

Volumetric Method for Methoxyl and Ethoxyl Estimations. A. Klemenc.

(*Monatsh. für Chem.*, 1913, **34**, 901-912.)—As a simplification in the estimation of methoxyl and ethoxyl groups by Zeisel's method, the author proposes a volumetric process based on the decomposition of the methyl or ethyl iodide by heat and titration of the iodine thus produced. The formation of alkyl iodide is effected exactly in the ordinary way in the small flask shown on the left of the diagram (see fig.). The vapours of iodide are carried forward by a current of air purified by potash lye, through a condenser 12-14 cm. long, and thence down a tube 7 mm. in diameter, into the washing flask, containing water and pure phosphorus. From this the alkyl iodide is conducted into the decomposition tube, the end of which should fit closely to the branch tube of the washing vessel in order to avoid contact of the vapours with the rubber joint. The tube is 25-28 cm. long, of hard Jena glass, having the shape illustrated in the diagram. It is charged with pieces of pumice, platinum strips, and platinised asbestos. The iodine liberated by the decomposition is sublimed over into the receiver flask containing a small cylinder into which the point of the delivery tube dips; this cylinder is charged with a concentrated solution of potassium iodide, whilst the flask itself and the bulb-attachment contain water. The decomposition tube has a diameter of 1·28 cm., and contains, at the end nearest the absorption flask, 1 grm. of 25 per cent. platinised asbestos; then follows a roll of platinum foil slit into narrow strips, kept in position by another plug of platinised asbestos, and the rest of the tube is filled with pieces of pumice held in position by an asbestos plug. Besides iodine, a little hydriodic acid is formed by the decomposition of the alkyl iodide. The liquid collected in the absorption flask is first titrated with $\frac{N}{10}$ or $\frac{N}{20}$ thiosulphate solution, and the product is then treated with 5 c.c. of a 4 per cent. solution of potassium iodate. The hydriodic acid present liberates iodine from this, and a second titration is made after allowing half an hour for the completion of the reaction. In starting the estimation, the apparatus is charged according to Zeisel's directions; the burners under the decomposition tube are lighted at the same time as that under the glycerol bath, and a moderate current of air is passed through. When the hydriodic acid begins to boil, the alkyl iodide is driven over with a slow current of air, and iodine begins to sublime at the cool end of the decomposition tube. It is important that the methyl iodide should enter the decomposition tube largely diluted with air. After the iodine vapours cease to form, the heating of the flask is discontinued, and a rapid stream of air is passed through while the decomposition tube is still heated for ten minutes. Finally, any iodine remaining in the delivery tube is dissolved by sucking back some of the potassium iodide solution, and the whole is then washed down. The titration of the hydriodic acid formed at the same time as the iodine, after the addition of potassium iodate, must be controlled by a blank titration with the same amounts of water and reagents. This is necessary because any carbon dioxide in the water and impurities in the reagents may liberate iodine from the mixture of iodide and iodate. Errors will arise if the decomposition of the alkyl iodide by heat be incomplete, also if any combustion take place with production of carbonic acid. Ordinarily, the limits of error lie between



+0·5 and −1·0 per cent. For the estimation of methoxyl, a tube charged only with pumice may be used, but for ethoxyl the platinum is necessary, owing to the formation of unsaturated hydrocarbons. J. F. B.

Rapid Volumetric Method for Estimation of Fatty Acids in Soaps. O. Schütte. (*Seifensieder-Zeit.*, 1913, **40**, 551-553; through *Chem. Zentralbl.*, 1913, II., 387.)—The method is analogous to the Gerber centrifugal method for the estimation of fat in milk. The special graduated tubes, which are made by Albert Dargatz, Hamburg 1, are so constructed that any volume of fat from 1·9 to 2·4 c.c. can be measured with an error not exceeding 0·01 c.c. A weighed quantity of the soap is dissolved in water and the solution made up to 500 c.c., the amount of soap taken being such that 25 c.c. of the solution will give between 1·9 and 2·4 c.c. of fatty acids. To 25 c.c. of the solution contained in the graduated tube, dilute (1 : 1) sulphuric acid is added drop by drop, the tube is placed in a hot-water-bath until the fatty acids separate as a clear layer, more acid is added to bring the fatty acid layer to the graduated part of the tube, and the latter is then whirled in a centrifuge. The tube is again warmed in a water-bath and the volume of the fatty acids layer read off. For purposes of works control, where tolerably uniform material is being used, the method gives excellent results with a minimum of trouble, as the density of the fatty acid layer may be assumed to be almost a constant. With soap of unknown origin, this density would need to be determined in each case. G. C. J.

Oxidation of Fatty Acids and Unsaponifiable Matter During Process of Fat Estimation by the Kumagawa-Suto Method. M. Tamura. (*Biochem. Zeitsch.*, 1913, **51**, 463-484.)—No change in weight or iodine absorption occurs on saponifying animal matter at 100° C. in a vessel covered with a clock glass, or in the evaporation of the petroleum ether solution of fatty acids by means of oxygen or air currents. On drying the residue after evaporation of petrol in an air oven at 50° C., an increase in weight and decrease in iodine absorption occurs, and hence it would be theoretically correct to dry in vacuo after expelling the petrol. If the duration of drying does not exceed one to two hours, this error may be neglected. The unsaponifiable matter also suffers no change during evaporation of solvent, but when dry is very sensitive at higher temperatures and loses in weight. The proposal of Mottram (*J. Physiol.*, 1909, **38**, 281; 1910, **40**, 122), that all operations should be conducted in the absence of air, is therefore unnecessary. E. W.

Analysis of Hardened Oils. W. Normann and E. Hugel. (*Chem. Zeit.*, 1913, **37**, 815.)—The acid value, saponification value, and proportion of unsaponifiable matter, are not materially affected by hydrogenising oils, but the hydroxyl value is reduced. Thus in the case of a sample of castor oil it fell from 156 to 102 and 131 in two experiments. In applying the method of Kreis and Roth (ANALYST, 1913, 160) to the detection of arachidic acid in hardened rape and marine animal oils, it is unnecessary to wait for three hours after the treatment with lead acetate, but only till the liquid has cooled to the ordinary temperature. As a rule the amount of fatty acids will not dissolve in the prescribed quantity of alcohol. They are therefore warmed with 100 to 150 c.c. of alcohol until dissolved, the solution cooled in water, and the deposit twice recrystallised from smaller quantities of alcohol, and its melting-point determined. Should this exceed 70° C., there can be no doubt that hardened arachis, rape, or marine animal oils, are present. But a lower melting-point does not necessarily prove the absence of those oils, since seal oil, for example, yields fatty acids, melting at 69° to 70° C. The cholesteryl acetate test may be used to distinguish between marine animal oils and rape or arachis oils (*cf.* ANALYST, 1913, 373). C. A. M.

Colour Reactions of Hardened Whale Oil. C. Grimme. (*Chem. Rev. Fett Ind.*, 1913, **20**, 155-156.)—Specimens of whale oil hydrogenised to different degrees were dissolved in a mixture in equal parts of benzene and xylene, and shaken with the usual reagents. It was found that the intensity of the colorations decreased with the degree of hardening, but that in each case it was possible to detect the presence of a marine animal oil. In particular, the test with 1 c.c. of sulphuric acid and 1 drop of tincture of iodine gave a very characteristic violet-red coloration in every instance. C. A. M.

Oil of Cohune Nuts. (*Bull. Imp. Inst.*, 1913, **11.**, 226-230.)—The nuts of the cohune palm (*Attalea cohune*), which is indigenous to British Honduras, contain a kernel which yields a white solid fat resembling cocoa-nut oil in appearance and odour. Five samples examined yielded 65·4 to 71·6 per cent. of fat with the following values: Sp. gr. at 100°/15·5° C., 0·868 to 0·871; acid value, 1·2 to 20·4; saponification value, 252·4 to 256·5; iodine value, 11·0 to 13·7; Hehner value (1 sample), 87·7; Reichert-Meissl value, 6·8 to 8·3; Polenske value (2 samples), 12·5 and 15·4; solidification point of fatty acids, 19·7° C. to 21·0° C.; and unsaponifiable matter, 0·23 to 0·28 per cent. The outer fibrous layers of the fruit also contained a dark green fat (9·3 to 20·6 per cent.), which could be partially decolorised by animal charcoal. A sample of the light greenish-yellow fat thus obtained gave the following results: Sp. gr. at 100°/15·5° C., 0·848; acid value, 162·0; saponification value, 197·4; iodine value, 75·1; Hehner value, 97·0; Reichert-Meissl value, 1·65; solidification point of fatty acids, 31·0° C.; and unsaponifiable matter, 0·95 per cent. C. A. M.

Colour Reactions of Oils with Certain Phenols in the Presence of Nitric Acid. M. Malacarne. (*Giorn. Farm. Chim.*, 1913, **62**, 153-169; through *Chem. Zentralbl.*, 1913, I., 2183-2184.)—Results of an investigation carried out with the purpose of ascertaining whether other phenols can be employed in Bellier's resorcinol test for sesame oil are recorded, particular attention being paid to the detection of seed oils in olive oil. The phenols used were dissolved in benzene when possible, but if not soluble were added in quantities of 0·01 grm. for each test. Each reaction was made in duplicate, in one case with nitric acid of sp. gr. 1·38, and in the other with the addition of acid of sp. gr. 1·20, the temperature of reaction being 30° C. in each instance. Only resorcinol and phloroglucinol yielded characteristic colour reactions with seed oils, whilst phenol, pyrocatechol, hydroquinone, hydroxyquinone, pyrogallol, α-naphthol and β-naphthol gave practically no coloration. The following colorations were obtained with different oils after the addition of resorcinol or phloroglucinol, and either the concentrated or the more dilute nitric acid mentioned: Olive oil: thirty-seven out of fifty samples examined gave a violet grey coloration, the acid layer being yellow; with the remaining thirteen samples a faint violet coloration was obtained. Sesame oil: a blue-violet or violet coloration, the acid layer being coloured green in the case of resorcinol and blue-violet with phloroglucinol. With earth-nut, cotton-seed, rape, radish, soya bean, linseed, maize, grape-seed, castor, almond and nut oils, a more or less intense violet coloration was obtained, the acid layer being yellow. It was found that the chromogen to which the coloration is due may be extracted to the extent of 80 per cent. by treating the oils with alcohol; it is not possible, however, to extract the substance completely. The unsaponifiable matters in the oils give the reaction, but not the fatty acids, the chromogen apparently being decomposed by alkalis. The colour reaction of sesame oil with resorcinol or phloroglucinol in the presence of nitric acid is so characteristic that it may safely be used for the detection of this oil in olive oil. W. P. S.

Detection of Rape Oil in Olive Oil. H. Kries. (*Chem. Zeit.*, 1913, **37**, 877-878.)—The fatty acids from 20 grms. of the oil are dissolved in 100 c.c. of 95 per cent. alcohol and the boiling solution is treated with 1·5 grm. of lead acetate dissolved in 50 c.c. of alcohol. After standing for twelve hours at a temperature below 15° C., the lead salts are collected, washed three times with alcohol, and the fatty acids are liberated from the salts by treatment with hydrochloric acid. The fatty acids thus obtained are again dissolved in 100 c.c. of alcohol, and the boiling solution is treated with 1 grm. of lead acetate dissolved in 50 c.c. of alcohol. After twelve hours the lead salts are separated by filtration, and the fatty acids remaining in the filtrate are recovered by evaporating off the alcohol and treating the residue with dilute hydrochloric acid. The melting-point of this fraction of the fatty acids is now determined. In the case of pure olive oil it will be about 47° C., but is considerably lower when rape oil is present. The method will detect the presence of as little as 5 per cent. of rape oil in olive oil. W. P. S.

Simple Reagent for the Detection of Mechanical Wood in Paper. E. Votoček. (*Chem. Zeit.*, 1913, **37**, 897.)—It has been observed that the tannin constituents of tea possess certain properties similar to those of phloroglucinol—for instance, they give greenish-black condensation products with furfural and 12 per cent. hydrochloric acid. Similarly, a decoction of tea may be employed as a simple, readily procurable substitute for phloroglucinol in testing paper for the presence of mechanical wood-pulp. A small quantity of tea, rich in tannin, is boiled for a short time with a few c.c. of water, the decoction is poured off or filtered and mixed with an equal volume of strong hydrochloric acid. If the solution is not clear, a little alcohol may be added. With a decoction of 1 grm. of tea in 25 c.c. of water, the violet coloration appears most rapidly with "Imperial" or "English" tea. With other teas it may require fifteen or thirty minutes to develop with full intensity. Tea decoctions also give with the vanillin-hydrochloric acid reagent the same coloration as phloroglucinol itself. The above reactions are not confined to the tannins of tea. They appear to be general with the tannins of the pyrocatechol type, whereas those purely of the pyrogallol type do not show the same reactions.

J. F. B.

Behaviour of Certain Sugars with Diphenylamine and Hydrochloric Acid. H. T. B. Rasmussen. (*Ber. deut. Pharm. Ges.*, 1913, **23**, 379-383; through *Chem. Zentralbl.*, 1913, II., 458.)—One c.c. of solutions of dextrose, galactose, mannose, lævulose, sorbose, cane-sugar, milk-sugar, maltose, arabinose, and xylose, was treated with 16 to 20 drops of a 10 per cent. alcoholic solution of diphenylamine, or a few crystals of the solid base, and 1 c.c. of fuming hydrochloric acid was then added. The mixture was boiled until a coloration appeared, and, after cooling, it was shaken with ether, whereupon the crystals which had separated were dissolved, and the liquid assumed a clear blue colour, whilst the ethereal layer, in the case of moderately concentrated sugar solutions, was coloured more or less greenish, probably owing to the formation of furfural. It was found that all hexoses give a more or less strong blue coloration with the diphenylamine and hydrochloric acid, the ketoses reacting more rapidly and strongly than the aldoses. The test is available for the detection of lævulose in urine, but not for the detection of lævulose or cane-sugar in the presence of several other sugars. Furfural gives a brown coloration with the reagent in the cold, which immediately becomes reddish-brown on heating, and subsequently almost black; on shaking out with ether the liquid becomes strongly green in colour. J. F. B.

I. Bangs' Method of Sugar Estimation. Z. Hatta. (*Biochem. Zeitsch.*, 1913, **52**, 1-44.)—Variation in the rate of titration may introduce an error of 5·2 to 6·8 per cent., and changing the temperature of titration from 10° to 30° C. may introduce a maximum error of 7·8 per cent. The most serious source of error is the reduction of the copper solution on boiling. The magnitude of this error varies with the sugar concentration, and may be as great as 9 per cent. on the positive side.

E. W.

Goldenberg Method (for Commercial Tartrates), 1898 and 1907. L. and J. Gadais. (*Bull. Soc. Chim.*, 1913, **13**, 722-725.)—The Goldenberg method has been widely adopted as a reference method in contracts for the sale of wine lees, tartar, etc. In 1907 the authors of the method suggested modifications both in the manner of carrying out the test and in computing the correction to be made for certain unavoidable errors (ANALYST, 1908, **33**, 200), and many analysts have adopted these suggestions. Others adhere to the earlier modification of the method known as the "Goldenberg method, 1898." The authors confirm the statement of others that, though on some samples the two methods yield identical results, in the great majority of cases the method of 1907 gives higher results. They think that the method of 1907 is to be preferred, as it is more elastic, yielding concordant results, even in the hands of two chemists whose methods of operation (*e.g.*, in the bulk of liquor used for washing) may differ very widely. But, as the older method seems unlikely to be abandoned in certain quarters, they point out that it might be made to yield more concordant results in the hands of different operators, if some restriction were placed on the amount of wash-water to be employed. G. C. J.

Quantitative Estimation of Trimethylamine in Presence of Ammonia. K. Budai. (*Zeitsch. Physiol. Chem.*, 1913, **86**, 107-122.)—The method is based upon the fact that trimethylamine does not react with formaldehyde. The ammoniacal nitrogen is estimated by treating the aqueous solution of the mixed hydrochlorides with an excess (10 c.c.) of formaldehyde (previously made neutral to phenolphthaleïn), and titrating with caustic alkali, using phenolphthaleïn as indicator. For the estimation of the total nitrogen, the liquid after titration is diluted with water, acidified with hydrochloric acid, boiled down to one-third its volume, made alkaline, and distilled into a known quantity of acid. E. W.

INORGANIC ANALYSIS.

Analysis of Brass. B. Koch. (*Chem. Zeit.*, 1913, **37**, 873-874.)—The following procedure is recommended for the electrolytic estimation of copper in brass. One grm. of the turnings is dissolved by heating in 10 c.c. of nitric acid of sp. gr. 1·20. One hundred c.c. of water and 15 c.c. of sulphuric acid (1 : 1) are then added, and the mixture is diluted with water to 150 c.c. The solution is now submitted to electrolysis at a temperature of 75° C., a current of 3·5 volts and 3 ampères being employed. The whole of the copper is deposited on the electrode within one hour under these conditions. With a current of 1 ampère and a temperature of 50° C. the time required is about two hours. W. P. S.

Behaviour of Calcium and Magnesium Salts with Soap Solutions, and the Determination of Hardness of Water. H. Masters and H. L. Smith. (*J. Chem. Soc.*, 1913, **103**, 992-998.)—It is impossible to obtain accurate measurements of hardness in water containing magnesium salts with a soap solution prepared from sodium oleate standardised with calcium salts. Solutions made from Castile soap are little better than solutions of pure sodium oleate. The difference in the behaviour of these solutions towards equivalent solutions of calcium and magnesium salts is shown to depend, in part at least, on the inferior solubility of magnesium oleate, particles of which separate, and tend to adsorb and carry down some sodium oleate. Making the determination at temperatures above that of the room, as suggested by Jackson (*Chem. News*, 1884, **49**, 149) and others, does not increase the accuracy of the results. Soaps prepared from saturated fatty acids are more satisfactory, but solutions of potassium palmitate, of the concentration necessary for carrying out Clark's test, tend to throw a precipitate of potassium hydrogen palmitate. Solutions of potassium myristate are free from this objection, and are consumed in exactly equal amount by equivalent weights of calcium and magnesium salts.

G. C. J.

Method of Estimating Carbon Dioxide. A. Dejeanne. (*Ann. Falsif.*, 1913, **6**, 335-339.)—In estimations of carbon dioxide, where the gas is absorbed in a definite quantity of barium hydroxide solution and the excess of the latter estimated at the end of the operation, it is recommended that magnesium chloride should be added when the absorption of the carbon dioxide is completed. The excess of barium hydroxide is thus converted into barium chloride, and an equivalent quantity of magnesium hydroxide is precipitated; this is removed, together with the barium carbonate, by filtration, and the barium chloride in the filtrate is estimated as sulphate. The addition of the magnesium chloride prevents the influence of atmospheric carbon dioxide during the subsequent filtration. W. P. S.

Examination of Portland Cement. B. Grünwald. (*Chem. Zeit.*, 1913, **37**, 885-886.)—The free magnesia in cement may be estimated as follows: The cement (0·5 grm.) is triturated in a mortar with 10 c.c. of water, and washed into the platinum-lined bomb of an Atwater calorimeter. The bomb is closed, filled with carbon dioxide under a pressure of 25 to 28 atmospheres, and agitated for six hours. The excess of gas is then allowed to escape, the bomb opened, and its contents filtered. Of the filtrate, an aliquot portion is titrated with $\frac{N}{10}$ hydrochloric acid, using methyl orange as indicator, and in the same portion the dissolved lime is determined gravimetrically. The equivalent of this lime is then deducted from the consumption of hydrochloric acid, and the balance is calculated to magnesia. G. C. J.

Detection of Chromium. A. Terni. (*Gazz. Chim. Ital.*, 1913, **43**, 63-65.)—On boiling the solution of a chromium salt with lead peroxide, in the presence of nitric, sulphuric, or hydrochloric acid (preferably the first), lead chromate is formed, and is kept in solution by the acid. On then decanting the liquid from the excess of lead peroxide (and lead sulphate in the case of sulphuric acid), and adding hydrogen peroxide and ether, the blue coloration of perchromic acid is obtained. In this way it is possible to detect 0·0000052 grm. of chromium. The method may also be used in examining the precipitate given with ammonia by metals of the third group. A small quantity of the precipitate is dissolved in nitric acid, and treated as described. If iron is present the decanted liquid is boiled with excess of sodium acetate, the iron being precipitated whilst the lead chromate remains in solution. For the detection of chromium in the presence of manganese the decanted nitric acid solution, which will be reddish-violet from the presence of permanganic acid, is heated with a few drops of hydrochloric acid, when the permanganic acid is decomposed, and the yellow coloration due to the chromate may be seen.

C. A. M.

Estimation of Hydroxylamine. E. Rupp and H. Mäder. (*Arch. der Pharm.*, 1913, **251**, 295-300.)—Meyeringh's method for the volumetric estimation of hydroxylamine by iodine in presence of a substance such as sodium phosphate or magnesia, to neutralise the hydriodic acid, suffers from two sources of error. In the first place, the neutralising agents combine with small proportions of iodine, and secondly, the reaction does not end at the stage at which nitrous oxide is evolved.

Of other known methods that of Raschig, according to which ferric sulphate is reduced by the hydroxylamine, and the ferrous salt titrated by permanganate, is the simplest and most accurate. The authors now describe two simple volumetric methods depending on the use of bromine as an oxidising agent. The bromine is best prepared from a solution containing 3·35 grms. of potassium bromate and 15 grms. of potassium bromide per litre; 25 c.c. of this solution, accurately standardised, equivalent to about 30 c.c. of $\frac{N}{10}$ thiosulphate solution, are diluted to about 100 c.c. in a roomy flask with glass stopper. A quantity of hydroxylamine solution containing not more than 0·01 grm. of hydroxylamine hydroxide is added, and then 10 c.c. of dilute sulphuric acid. The flask is allowed to stand stoppered for twenty to thirty minutes, 1 grm. of potassium iodide is added, and the iodine is titrated with $\frac{N}{10}$ thiosulphate solution after standing for a minute. The hydroxylamine is oxidised by bromine to nitric acid, according to the equivalent proportions 1 $NH_2OH = 6\,Br$, so that 1 c.c. of $\frac{N}{10}$ thiosulphate represents 0·00055 grm. of hydroxylamine hydroxide. The conditions necessary to obtain accurate results are only a very moderate acidity and a good excess of bromine. Incomplete reactions are shown by a rapid return of the colour after titration is completed. Another modification of the same reaction depends on the use of sodium hypobromite, which must, however, contain no free alkali. For preparing the reagent, 10 grms. of caustic soda are dissolved in a litre of water, and 15 grms (5 c.c.) of bromine are added. For standardising the solution, 20 c.c. are placed in a stoppered flask with 50 c.c. of water, 1 grm. of potassium iodide is added, and lastly 20 c.c. of dilute hydrochloric acid; the iodine is then titrated with $\frac{N}{10}$ thiosulphate. For the estimation, 20 c.c. of the hypobromite solution are placed in the flask with 75 c.c. of water, and dilute hydrochloric acid is added drop by drop until the pale yellow colour of the solution just changes to reddish-brown, indicating the neutralisation of the alkali. A quantity of hydroxylamine not exceeding 0·01 grm. of the hydroxide is then added, and the mixture allowed to remain for five minutes. After the addition of potassium iodide and acid the titration may be completed with thiosulphate. The reagents should be adjusted so that about one-half of the hypobromite taken remains in excess. J. F. B.

Estimation of Hypophosphites. W. D. Cocking and J. D. Kettle. (*Paper read before Brit. Pharm. Conf.*, 1913; through *Pharm. J.*, 1913, **91**, 132-134.)—The hypophosphite is oxidised to phosphate with potassium bichromate and sulphuric acid after precipitation of any phosphite with lead acetate, and the excess of bichromate titrated. A solution of 2·5 grms. of the salt in 40 c.c. of water is treated with excess (about 5 c.c.) of 10 per cent. lead acetate solution, and then made up to 50 c.c., shaken, and allowed to stand for about an hour. Ten c.c. of the clear supernatant liquid are heated for an hour on the water-bath with 50 c.c. of $\frac{N}{1}$ potassium bichromate solution and 10 c.c. of sulphuric acid, then diluted to 250 c.c., and 50 c.c. treated with potassium iodide and titrated with $\frac{N}{10}$ sodium thiosulphate solution. Phosphites are estimated by repeating the oxidation without the preliminary treatment with lead acetate, and calculating the difference between the two results into phosphite. In the examination of commercial samples by this method the following results were obtained: Calcium hypophosphite

(6), 95·95 to 98 per cent.; moisture, 0·05 to 0·1 per cent., with traces of sulphate. Sodium hypophosphite (8), 84·5 to 94·5; moisture, 5·7 to 11·1 per cent.; phosphites, a trace. Potassium hypophosphite (2), 94·5 and 99·5 per cent. Manganese hypophosphite (6), 89·3 to 103·5 per cent. Ferric hypophosphite, 73·7 to 90·8 per cent. In the case of ferrous hypophosphite the amount of ferrous iron is estimated by dissolving the salt in hydrochloric acid and pouring the solution into a large excess of hot mercuric chloride solution. Mercurous chloride is precipitated, and the hypophosphite oxidised to phosphate, after which the ferrous iron is titrated with potassium bichromate solution. In a second estimation the salt is oxidised with bichromate, the excess of which is subsequently titrated with ferrous ammonium sulphate, the result corresponding to the hypophosphite and ferrous iron together. Phosphite is included with hypophosphite in the case of iron salts. Commercial ferrous hypophosphite consists of variable proportions of ferrous and ferric salts. Thus two samples contained 33·76 and 44·99 per cent. of the former, and 47·82 and 24·37 per cent. of the latter, respectively. The former also contained 6·71 per cent. of sodium hypophosphite and 7·23 per cent. of sodium sulphate, and the latter 22·65 per cent. of calcium hypophosphite. C. A. M.

Mercuric Oxide as a Standard for Volumetric Analyses. L. Rosenthaler and A. Abelmann. (*Paper read before Brit. Pharm. Conf.*, 1913; through *Pharm. J.*, 1913, **91**, 144-145.)—Mercuric oxide, being readily obtained pure in commerce and not altering in composition if protected from light, is suitable for standardising volumetric solutions. As a standard in acidimetry, a weighed quantity of mercuric oxide is dissolved on the water-bath in excess of $\frac{N}{10}$ hydrochloric acid (which has been standardised on sodium oxalate) with a sufficient quantity of neutral sodium chloride, and the excess of acid titrated back with $\frac{N}{10}$ sodium hydroxide solution, with *p*-nitrophenol or iodeosin as an indicator. As a standard for iodimetry, the mercuric oxide is dissolved in hydrochloric acid, and sufficient potassium iodide added just to redissolve the precipitate first formed. The solution is next made alkaline with 10 to 20 c.c. of 10 per cent. potassium hydroxide solution, and the flask rotated while a mixture of about 3 c.c. of pure 35 per cent. formaldehyde solution and 10 c.c. of water is added, to precipitate metallic mercury from the double iodide. After three minutes' shaking, the solution is acidified with dilute acetic acid, again shaken, and treated with an excess of $\frac{N}{10}$ iodine solution until no more mercury remains undissolved. The uncombined iodine is then titrated with sodium thiosulphate solution. For standardising oxidising solutions, the mercuric oxide is reduced with formaldehyde as described, the mercury collected on a filter of asbestos and glass-wool, washed, and converted into mercuric sulphate by means of potassium permanganate and sulphuric acid, and the excess of permanganate titrated with oxalic acid. Or, instead of formaldehyde, potassium arsenite may be used for reducing the mercuric oxide, the mercury in this case being more easily washed and dissolved in the permanganate solution. Mercuric oxide may also be used as a standard for ammonium thiocyanate solutions. A weighed quantity is dissolved in nitric acid, and the solution treated with 3 c.c. of concentrated nitric acid and about 3 c.c. of a saturated solution of ferrous ammonium sulphate, and titrated with $\frac{N}{10}$ ammonium thiocyanate solution. C. A. M.

Estimation of Small Quantities of Lead. A. V. Elsden and J. F. Stansfield. (*J. Chem. Soc.*, 1913, **103**, 1039-1042.)—The method depends on the concentration of the lead by precipitation with ammonia in presence of ferric iron (*cf.* Wilkie, ANALYST, 1909, **34**, 414), and the subsequent separation of lead from iron by precipitation as lead molybdate in a solution in which the iron exists as a ferrous salt, which is not precipitated on boiling the weak acetic acid solution in which alone the precipitation of lead molybdate is complete. To the solution is added at least ten times as much iron (as ferric chloride) as there is likely to be lead present. Ammonia is added in excess, and the solution boiled and filtered. The precipitate is dissolved on the filter by means of 10 to 20 c.c. hot hydrochloric acid, followed by hot water, until about 75 c.c. has been collected. Ammonia is added to the solution until a slight permanent precipitate results, and then $\frac{N}{2}$ hydrochloric acid is added at the rate of 2 c.c. per decigram of iron, with a minimum of 10 c.c. Sodium sulphite is added at the rate of 0·2 grm. per decigram of iron, with a minimum of 1 grm., and the solution warmed until reduction is complete. After addition of 10 to 20 c.c. of 10 per cent. ammonium acetate solution the mixture is heated to boiling, and the lead precipitated by the slow addition of 10 to 20 c.c. ammonium molybdate solution (5 per cent.). After boiling a short time the precipitate is filtered off, and washed with boiling water. As it always contains a little iron, it is redissolved on the filter by means of 10 c.c. hot hydrochloric acid, followed by hot water, until 50 c.c. or less has been collected. A few drops of tartaric acid solution and of litmus solution are added, the solution is neutralised by means of strong ammonia, and then acidified with a few drops of $\frac{N}{2}$ hydrochloric acid. A few c.c. of ammonium acetate solution are added, the mixture boiled, and the purified lead molybdate is filtered off, washed, ignited with the paper, and weighed. Brearley (*Chem. News*, 1898, **78**, 203) has shown that many metals do not interfere with the determination of lead as molybdate. Tin, antimony, and phosphorus, if present, are carried down by the iron precipitate, which should in such cases be dissolved in hydrochloric acid, after which lead, tin, and antimony can be separated from iron and phosphorus by means of hydrogen sulphide, and the lead sulphide freed from the sulphides of tin and antimony by treatment with ammonium sulphide. The lead may then be conveniently determined by dissolving the sulphide in hydrochloric acid containing bromine and precipitation as molybdate as described. Quantities of lead of the order of 1 mgrm., in such materials as brass, phosphor-bronze, etc., can be determined with an error not exceeding +0·1 mgrm., and quantities of the order of 10 mgrm. with an error not exceeding + 0·3 mgrm.

G. C. J.

Estimation of Nitrites by Means of Thiocarbamide, and the Interaction of Nitrous Acid and Thiocarbamide in Presence of Acids of Different Strength. M. E. Coade and E. A. Werner. (*J. Chem. Soc.*, 1913, **103**, 1221-1228.)—When a nitrite is added to an acid sólution of thiocarbamide, the reaction may proceed in two directions, depending on the strength of the acid. Thus, in presence of acetic acid, the reaction takes place mainly in accordance with the equation—

$$CSN_2H_4 + HNO_2 = HSCN + N_2 + 2H_2O,$$

whilst in presence of hydrochloric acid the reaction—

$$2CSN_2H_4 + 2HNO_2 = C_2S_2N_4H_6 + 2NO + 2H_2O$$

predominates. Since the volume of gas produced by a definite weight of nitrous acid is the same whichever course the reaction takes, the fact that neither reaction can be wholly repressed is of no importance so far as the gasometric determination of nitrites is concerned. The use of acetic acid is preferred because nitrogen is less soluble than nitric oxide in water. A quantity of the nitrite, corresponding with a milligram molecular proportion of nitrous acid, is dissolved together with 0·1 grm. thiocarbamide in 1 c.c. of water in the cup of a nitrometer, washed in with the aid of another 0·5 c.c. water, and followed by 1 c.c. of 20 per cent. acetic acid. In about two minutes the reaction is complete, and the volume of gas can be read off. As compared with the carbamide method, there is no absorption of carbon dioxide to make, thus reducing the chances of error. The volume of gas can be read off with great exactness, as the surface of the mercury remains clean after repeated use, which is in marked contrast to what occurs when using the potassium iodide method. The results with pure silver nitrite are exact. Commercial sodium nitrite was found to contain 96·75 per cent. of the pure salt, a result in exact agreement with others obtained by Rupp's permanganate method (*Zeitsch. anal. Chem.*, 1906, **45**, 690), whereas the carbamide method gave only 92·9 per cent. Solutions of ethyl nitrite in alcohol appeared weaker when tested by the thiocarbamide method than when tested by the potassium iodide method, and the difference was most marked (10 per cent. on the ethyl nitrite) in the case of old solutions known to contain ethyl nitrate. The thiocarbamide method is shown to be accurate in presence of ten times as much nitrate as nitrite. G. C. J.

Estimation of Active Oxygen in Perborates and in Washing Powders containing Perborates. F. M. Litterscheid and P. S. Guggiari. (*Chem. Zeit.*, 1913, **37**, 677-679; 690-691.)—Numerous methods have been proposed for the estimation of active oxygen in perborates and other per-salts, and, having investigated many of these methods, the authors come to the conclusion that any and all of the following processes are trustworthy: Of the volumetric methods, titration with arsenious acid (*cf.* ANALYST, 1900, **25**, 136; 1902, **27**, 131), with permanganate (*ibid.*, 1910, **35**, 372), or the iodimetric process, may be recommended. In the case of washing powders, the sample should be dissolved in water, the solution acidified with sulphuric acid, diluted to a definite volume after the addition of a quantity of ignited kieselguhr, and filtered; the filtrate is employed for the estimation. This way of treating washing powders is also followed when the active oxygen is estimated gasometrically or gravimetrically. For the gasometric estimation the method described by Bosshard and Zwicky (*ibid.*, 1910, **35**, 372) may be used, but it must be remembered that one-half of the volume of oxygen evolved is derived from the manganese dioxide, the other half coming from the perborate. The quantity of oxygen evolved when a perborate is treated with sulphuric acid in the presence of manganese dioxide may also be estimated gravimetrically, an ordinary carbon dioxide apparatus being employed. W. P. S.

Use of Sodium Potassium Tartrate in the Estimation of Dissolved Oxygen in Water. J. J. van Eck. (*Chem. Weekblad*, 1913, **10**, 455-464; through *Chem. Zentralbl.*, 1913, II., 304-305.)—The addition of sodium potassium tartrate has been recommended in this estimation for the purpose of preventing the formation of a precipitate, but the addition in the case of waters containing large quantities of magnesium salts causes the results for dissolved oxygen to be too low. The author shows that this deficiency is due to the production of various basic manganese tartrates and manganese sodium tartrates. These dissociate and absorb oxygen, and may subsequently decompose the potassium iodide with liberation of iodine in the final titration. The difficulty may be overcome to some extent by the addition of larger quantities of sodium hydroxide and the other reagents. W. P. S.

Rapid Determination of Total Carbon in Steel, Cast Iron, and Iron Alloys. H. de Nolly. (*Chem. Engineer*, 1913, **18**, 30-32.)—The method depends on the combustion of the iron in a limited volume of oxygen, absorption of the carbon dioxide in a definite volume of $\frac{N}{10}$ caustic alkali, and titration of the alkaline solution with standard acid in presence of phenolphthaleïn. The combustion is carried out in a 1,000 c.c. conical flask of Jena glass. The neck of the flask is closed by a three-hole rubber bung protected by asbestos. Through two of the holes pass insulated copper electrodes which extend about half-way down the flask. The third hole accommodates a copper tube which is connected with a supply of oxygen under a pressure of about 12 to 16 inches of water. Attached to this copper tube is a stout copper support for the asbestos cup, which serves to hold the finely divided metal, and below the asbestos cup is a wire cage or clay cup to catch any molten oxide, should the asbestos cup be damaged. The ends of the electrodes just touch the metal in the cup. The flask is filled with oxygen, a measured quantity of $\frac{N}{10}$ caustic alkali introduced, the stopper bearing the electrodes and cup of finely divided metal is set in its place, and connection made with the oxygen gas-holder, between which and the apparatus is a mercury seal to prevent any blowing back. A 12 to 15 ampère arc is then set up between the ends of the electrodes to initiate the combustion, after which the circuit is broken and the combustion allowed to proceed. Occasionally it may be necessary to re-establish the arc or to moderate the supply of oxygen. Unless the metal is very finely divided, it may be necessary to mix it with 20 to 100 per cent. of its weight of lead peroxide, and if the carbon content be very high, to dilute it with extra-mild steel of known carbon content. When combustion is complete, the stopper is quickly replaced by a solid bung, and the contents of the flask are shaken and titrated. The whole series of operations requires less than ten minutes, and the results are said to be highly satisfactory for controlling the operation of a Siemens-Martin or electric furnace, but no test numbers are given. G. C. J.

Rapid Method for Determination of Phosphorus in Steel. H. Bhattacharyya. (*J. Soc. Chem. Ind.*, 1913, **32**, 738-739.)—A modification of the method which depends on solution of the well-washed phosphomolybdate precipitate in a definite amount of caustic alkali and titration of the excess of alkali in presence of phenolphthaleïn. The modification consists in the use of a 1 per cent. solution of potassium nitrate to wash out the 1 per cent. nitric acid with which the precipitate is first washed. G. C. J.

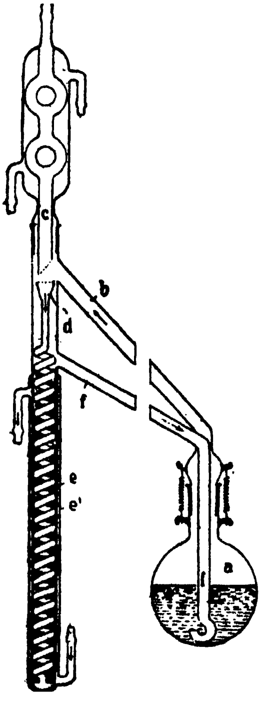
Separation of Thorium from Monazite Sand by Means of Hypophosphorous Acid. Detection of Cerium. F. Wirth. (*Chem. Zeit.*, 1913, **37**, 773-774.)—The use of alkali hypophosphites for the separation of thorium from sulphuric acid extracts of monazite sand has the drawback that insoluble alkali double sulphates of the cerium earths may simultaneously be produced. This is obviated by causing the anion of hypophosphorous acid to be formed directly in the acid extract. For this purpose the best results are obtained by anodic oxidation of copper phosphide in the extract, after preliminary neutralisation of the free acid to the point at which the phosphates are still kept in solution. Then, under the influence of a current of 10 volts the ions $[PO_3]''$ and $[HPO_3]'$ are formed, and these immediately combine with the thorium ions to produce insoluble thorium hypophosphite. The precipitation can also be effected in alkaline solution. For example, if a slightly ammoniacal solution of thorium tartrate be boiled with a large excess of sodium hypophosphite, the whole of the thorium is precipitated, provided that the liquid be kept alkaline during the boiling.

Detection of Cerium.—The neutral solution to be tested is boiled with 5 c.c. of a 10 per cent. solution of ammonium tartrate and 5 c.c. of dilute ammonia solution, when a yellow to brown coloration (due to oxidation) is obtained if cerium is present. Hydrogen peroxide precipitates cerium peroxide from concentrated alkaline cerium tartrate solutions, whilst in dilute solutions the coloration produced will detect 0·0002 grm. of cerium oxide in 100 c.c. C. A. M.

Estimation of Zinc as Zinc Ammonium Phosphate and Zinc Phosphate. T. M. Finlay and A. C. Cumming. (*J. Chem. Soc.*, 1913, **103**, 1004-1010.)—The modification of the phosphate method described in Low's *Technical Methods of Ore Analysis* (1911 edition, p. 301) sometimes leads to results 20 per cent. below the truth, and that in spite of the fact that the precipitate always contains sodium derived from the microcosmic salt employed. The low results are due to the solubility of the precipitate in the comparatively high concentration of acetic acid permitted by Low. Dakin's modification (*Zeitsch. anal. Chem.*, 1901, **39**, 273) yields accurate results provided (1) the solution be not alkaline and (2) no free mineral acid nor more than a trace of acetic acid be present. Moreover, since it is difficult to wash out all the soluble salts of ammonia when these are present in quantity, as Dakin himself observed, the authors express the opinion that the precipitate should always be ignited to pyrophosphate without attempting to dry at 105° C. and weigh the zinc ammonium phosphate. The examination of Low's method directed attention to another possible source of error, namely, the formation of double phosphates of zinc and the alkali metals in solutions containing large quantities of salts of sodium and potassium. The influence of sodium salts can be entirely overcome by addition of a considerable quantity of ammonium chloride, but a second precipitation is necessary when potassium salts are present.

As commercial "ammonium phosphate" is not always the same salt, the authors add the phosphate to the acid solution of zinc and then adjust the acidity. The acid solution of zinc (150 c.c.) is warmed on the water-bath, about 1 grm. of ammonium phosphate is added for each decigram of zinc probably present, the solution is neutralised with ammonia and then acidified by addition of 1 c.c. of 2N acetic acid. If salts of the fixed alkalis are probably present, from 5 to 10 grms. of ammonium chloride must be added before the ammonium phosphate. After fifteen minutes on the water-bath, the precipitate will have assumed a crystalline form, and the beaker is then set aside for its contents to cool. In absence of potassium salts, the precipitate is filtered off on a Gooch crucible, washed first with 1 per cent. ammonium phosphate, then with water, and finally ignited and weighed as zinc pyrophosphate. In presence of potassium salts, the precipitate is filtered, washed with dilute ammonium phosphate, and redissolved in hydrochloric acid. From 5 to 10 grms. ammonium chloride is added to the solution, which is then neutralised and the zinc is precipitated as before, and this time the precipitate is thoroughly washed and weighed as zinc pyrophosphate. The extreme errors recorded are −0·3 mgrm. and +1 mgrm. on quantities of 0·2 grm. of zinc. G. C. J.

APPARATUS, ETC.



Apparatus for Extracting Liquids Decomposed by Heat. R. Kempf. (*Chem. Zeit.*, 1913, **37**, 774.)—The liquid to be extracted is placed in the cylindrical vessel, *e*, which is surrounded by a cooling jacket, e^1. The solvent is heated in the flask *a*, and the vapours passing through *b* are condensed in the cooler *c*, and fall into the funnel *d*, the extremity of which ends in a long spiral. Issuing from the base of this, the solvent rises through the liquid and the extract passes through the tube, *f*, back into the flask *a*. This apparatus may be used for the quantitative estimation of tannin in ink by extraction with ethyl acetate, which when heated is readily decomposed by the hydrochloric acid in the ink. C. A. M.

Improved Apparatus for Extracting Finely Divided Solids with Volatile Solvents. M. Kardos and W. Schiller. (*Chem. Zeit.*, 1913, **37**, 920.)—The improvement seeks to overcome the tendency of the central portion of the contents of an extraction thimble to escape the action of the solvent. The apparatus used is the ordinary Soxhlet extractor and paper thimble, with the addition of a distributor which occupies the position of the central core of substance which, in the ordinary way, tends to escape the action of the solvent. This distributor is a stout glass tube, somewhat longer than the thimble, closed at the lower end, and flared to a funnel shape at the top so as to catch the whole of the solvent as it drops from the condenser. Throughout its length the distributor is pierced by numerous pin-holes, and these are prevented from being choked by a close-fitting sheath of linen which covers the distributor from its lower end up to the flared top. The distributor is placed centrally in the thimble, and the material to be extracted is packed round it. Test numbers show that extraction is much more rapid with the new apparatus than without it. G. C. J.

Apparatus for Use in the Estimation of Starch by Ewers' Process. C. Grimme. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1913, **25**, 726-727.)—The apparatus consists of a circular holder in which may be fitted eight flasks containing mixtures of starch and acid as prescribed in Ewers' method (ANALYST, 1908, **33**, 101, 481); the holder is provided with a gauze cover and a handle so that the flasks may be immersed together in a boiling water-bath, and removed therefrom when the period of heating is at an end. W. P. S.

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REVIEW.

CHEMISTRY OF THE OIL INDUSTRIES. By J. E. SOUTHCOMBE, M.Sc. London: Constable and Co., Ltd. 1913. Pp. 204. Price 7s. 6d. net.

This book forms one of a series of "'Outlines of Industrial Chemistry' . . . Introductory to the Chemistry of the National Industries." Edited by Guy D. Bengough, M.A., D.Sc.

The author points out "that in so small a volume it has been impossible to deal in any way completely with the multitude of facts and theories relative to such an extensive subject; and the object of the work is rather to fill the gap between the elementary textbooks of pure chemistry and the numerous technical treatises and monographs of a highly specialised character."

The wide scope of the work will be realised when one considers that it touches both on the mineral and fatty oil industries, either of which provides a very extensive subject and one difficult to compress.

In the first chapter the student is led by easy stages from the simple hydrocarbons to the more complicated bodies found in oils, fats, and waxes. The chemical terms are not always in the most approved form, but one cannot justly take exception to departures from orthodoxy when they aid lucidity.

Chapter II. deals with mineral oils, their source, fractional distillation, and methods of refinement. The next three chapters outline the methods of preparation and refinement of saponifiable oils and fats and embody notes on their sources, properties, and general composition.

Chapters VI. and VII. consist for the most part of sections dealing with eleven vegetable oils and fats, together with the more important animal fats, fish oils, and natural waxes. Tables of chemical and physical constants are given, but these are rather brief, and might with advantage have included some refractive indices, of which none are to be found, although the Abbé refractometer is described, with a full-page illustration, under "Analytical Methods."

Chapter VIII., on analytical methods, must be taken rather as indicating some of the methods usually employed than as providing a full guide to analytical procedure, as, with the exception of some of the more common determinations, the methods savour more of the lecture-room than of the technical laboratory. In the case of the isolation of unsaponifiable matter, for instance, 1 to 2 litres of ether seem to be necessary.

The remainder of the book deals for the most part with the industrial application of oils and fats. In the section on soap-making the author has attempted to elucidate the scientific principles underlying the manufacturing operations with the help of the phase rule and theories of adsorption. His views on colloidal chemistry do not appear to be in agreement with those generally held, and would probably lead to misconception. Rewritten in a fuller and more correct form, they would be valuable to the student.

The book is readable and abounds with technical terms used in manufacture, with very correct interpretations, but there is throughout the work a looseness of expression and a want of consistency in chemical nomenclature (such as "phytosterine," "phytostearol," "phytosterol acetate") which might easily be rectified.

The book is undoubtedly useful as containing a series of "stepping-stones," but the student is warned that some of them are rather slippery and his knowledge should eventually rest on a fuller and firmer foundation than this work supplies.

E. R. Bolton.

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THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

THE ESTIMATION OF IRON IN SYR. FERRI PHOSPH. CO. AND OTHER PHARMACEUTICAL PREPARATIONS.

By NORMAN EVERS, B.Sc., F.I.C.

Syr. Ferri Phosph. Co.—The estimation of iron in chemical foods is attended by many difficulties. The ordinary precipitation or titration cannot be carried out in the presence of the sugar and cochineal; and, further, the removal of the organic matter by ignition or oxidation is tedious, and it is not always easy to dissolve the residue obtained.

A process has been recently put forward by Salamon and Seaber (*Chemist and Druggist*, 1913, **83**, 2) for the estimation of total iron in *syr. ferri phosph. co.* depending on precipitation with ammonia, washing out the sugar, dissolving the precipitate in dilute acid, reducing, and titrating with permanganate. In my experience it was not possible to remove the sugar in one precipitation, even by prolonged washing with boiling water. High results were therefore obtained.

Recently, having had to estimate the iron in this preparation, I used the following method, depending on oxidation of the iron with nitric acid, and direct colorimetric estimation as thiocyanate. This procedure has the advantage of being rapidly and easily carried out, and is sufficiently accurate for ordinary purposes. Moreover, the iron present in the ferrous condition can also be determined.

Estimation of Total Iron.—Ten c.c. of the syrup are pipetted into a flask, diluted with 50 c.c. of water, 5 c.c. of strong nitric acid added, and boiled for about a minute to convert the iron into the ferric condition. The liquid is then cooled and made up to 100 c.c., 5 c.c. diluted to 100 c.c., and 5 c.c. of the diluted solution measured into a 100 c.c. Nessler cylinder, and diluted to the 50 c.c. mark with water. Five c.c. dilute hydrochloric acid (1 in 3) and 10 c.c. of 5 per cent. potassium thiocyanate solution are then added. The colour is compared with that formed under the same conditions by a standard solution of ferric chloride (1 c.c. = 0·00002 Fe), as in the process of Nesslerising. In the final comparison the thiocyanate should be added to the iron, not *vice versa*. In about ten minutes the colour begins to fade slightly; comparisons should therefore be made within that time. Using the above quantities, 1 c.c. standard iron solution = 0·08 per cent. iron in the syrup. As the addition of 0·1 c.c. standard iron produces a distinct change in the depth of colour, the process is accurate to 0·008 per cent. of iron, which is sufficient for ordinary purposes.

The process has been tested, using known quantities of ferrous iron, with the same proportions of sugar and cochineal as are present in the syrup. In each case the results obtained were within the limits of error of the process:

		Grm.	
Iron taken	0·0389	0·0220	0·0193
,, found	0·0388	0·0216	0·0194

Estimation of Ferric Iron.—Ten c.c. of the syrup are diluted to 100 c.c. with air-free water, 10 c.c. again diluted to 100 c.c. with air-free water, and 20 c.c. taken in a Nessler cylinder. The slight tinge of cochineal which still remains is matched with standard ferric chloride and thiocyanate, as it is of the same tint. Another 20 c.c. of the diluted solution is then taken, diluted to 50 c.c. with air-free water, 5 c.c. dilute hydrochloric acid and 10 c.c. thiocyanate added, and the colour matched as before. The number of c.c. of standard iron solution used to match the cochineal is subtracted from the final amount used.

One c.c. standard iron solution = 0·01 per cent. ferric iron in the syrup. In order to prove that no oxidation occurred on dilution, a similar quantity of ferrous iron was diluted under similar conditions. A mere trace of colour was obtained.

RESULTS.

Sample.	Total Iron per Cent.	Ferric Iron per Cent.	Ferrous Iron per Cent.
A	0·424	0·024	0·400
B	0·412	0·016	0·396
C	0·408	0·013	0·395
D	0·408	0·018	0·390
E	0·408	0·034	0·374
F	0·344	0·020	0·324
G	0·300	0·016	0·284
H	0·248	0·005	0·243

Sample A was carefully prepared by myself in accordance with the directions of the B.P. Codex. The other samples were from various commercial sources. Sample E had been kept for about a month, and had deposited somewhat, whence the high percentage of ferric iron. After standing a fortnight, the percentage of ferric iron in A had increased to 0·031 per cent.

The method has also been applied to the following preparations:

Syr. Ferri Phosph. c̄ Quinin. et Strychnin. (Easton's Syrup).—Five c.c. are taken and treated as above.

One c.c. standard iron solution = 0·16 per cent. Fe.

A syrup prepared containing 0·80 per cent. Fe gave 0·80 per cent. by this method. Other syrups gave the following results:

	A.	B.
Total iron	0·80	0·75
Ferric iron	0·02	0·02
Ferrous iron	0·78	0·73

Liq. Ferri Hypophosph. Fort.—The estimation of iron in this preparation is somewhat troublesome on account of the fact that, in order to precipitate the iron as hydroxide, it is necessary to use strong potash, which is difficult to remove by washing. It is necessary to dissolve the precipitate in acid after ignition, and reprecipitate with ammonia. The colorimetric process described above was used, leaving out the oxidation with nitric acid, diluting 1,000 times instead of 200, and taking 5 c.c. for comparison.

One c.c. standard iron solution = 0·4 per cent. Fe.

Owing to the larger amount of iron present, the error in this case is greater; but where time is important, and only approximate results are required, the process is useful. The following are the results:

	Iron per Cent.	
	A.	B.
Gravimetric	1·92	1·57
Colorimetric	1·96	1·60

Syr. Ferri Hypophosph.—In this case the colorimetric method is of great value, as the presence of the sugar interferes with the gravimetric estimation.

Ten c.c. of the syrup are diluted 200 times, and 5 c.c. taken for comparison. Two syrups prepared from the above solutions of ferric hypophosphite gave the following results:

	Iron per Cent.	
	A.	B.
Theory	0·384	0·314
Found	0·376	0·312

Mist. Ferri Co.—Ten c.c. are diluted to 50 c.c. with water, boiled for one minute with 5 c.c. nitric acid, cooled, diluted to 100 c.c., 10 c.c. of this diluted to 100 c.c., and 5 c.c. taken for comparison.

One c.c. standard iron solution = 0·04 per cent. Fe.

A prepared sample, containing 0·114 per cent. Fe, gave 0·116 per cent. Fe by this method.

These are all the cases in which the method has been tried, but obviously there are others to which it would be equally applicable. It appears to be especially useful where the amount of iron is less than 1 per cent., and the organic matter present is such as to prevent the usual estimation by titration or precipitation.

* * * * *

NITRATES AND NITRITES IN MILK.

By G. D. ELSDON, B.Sc., A.I.C., and JOHN A. L. SUTCLIFFE, A.I.C.

A portion of the matter contained in the present communication has received lately a considerable amount of attention. Our results, however, are in some respects contrary to those obtained by other workers, and we therefore publish them, thinking that they may be of general interest. We suggest also a new method for the determination of nitrates in milk when present in any considerable quantity, and which is applicable in the presence of nitrites.

As milk serum must be used in the processes for the estimation of nitrates in milk, we here describe the method of preparation we have adopted.

Preparation of Milk Serum.—We find that the best results are obtained by the method suggested by Tillmans and Sutthoff (*Zeitsch. Untersuch. Nahr. Genussm.*, 1910, **20**, 49; Analyst, 1910, **35**, 434), a slight modification being made for the sake of convenience and rapidity. The reagent consists of a 2·5 per cent. solution of mercuric chloride in 1 per cent. w/v hydrochloric acid, and we proceed as follows: Twenty-five c.c. of milk are mixed with 25 c.c. of the reagent, and after well mixing, the whole is poured on to an 11 cm. filter-paper and 25 c.c. of filtrate collected. The rate of filtration may be hastened very considerably by warming the milk before adding the reagent, but in this case the filtrate obtained is not quite so clear. We prefer to work in the cold.

The Detection of Nitrates in Milk.—Since milk is normally free from nitrates, their presence affords strong presumptive evidence of the addition of water, and therefore several methods have been suggested for their detection. The method that seems to have been most favoured is that which depends upon the blue coloration produced by a solution of diphenylamine sulphate in concentrated sulphuric acid. We have found this test to be both delicate and easy of application, and we therefore recommend it for the detection of nitrates; but as the reaction is also given by nitrites, it cannot be used in the presence of the latter. The solution before being examined for nitrates should always be tested for the presence of nitrites by the Griess-Ilosvay method. If nitrites are found, a special method has to be followed in order to test for nitrates. For this purpose we use the brucine-sulphuric acid reagent, which is described later.

We would here draw attention to the erroneous conclusions of P. Soltsien (*Pharm. Zeit.*, 1906, **51**, 765; Analyst, 1906, **31**, 374) concerning the diphenylamine test. He states that nitrous acid not only fails to give the reaction, but actually hinders it, when nitrous and nitric acids are present together. We have found that this is not the case, as nitrous acid not only does give the reaction, but gives a deeper colour in equivalent strengths than does nitric acid, and in a shorter time.

The Diphenylamine Test for Nitrates.—The test is carried out in the following manner, which is a slight modification of the method suggested by Tillmans (*Zeitsch. Untersuch. Nahr. Genussm.*, 1910, **20**, 676; Analyst, 1911, **36**, 67): The reagent is prepared by adding 50 c.c. of water to 0·085 grm. of diphenylamine sulphate contained in a large flask, and gradually adding 450 c.c. of strong sulphuric acid. In performing the test 1 c.c. of the serum of the suspected milk is placed in a test-tube (4 by 0·5 in.), 4 c.c. of the reagent are added, and the whole thoroughly mixed and cooled. The presence of traces of nitrates causes the production of a blue colour.

The Brucine-Sulphuric Acid Method.—In the presence of nitrites this reagent (see below) gives an immediate red coloration, which gradually fades to a pale straw tint in three to six hours if nitrates be absent. In the presence of nitrates *and* nitrites, however, although the first immediate deep red colour will fade, a reddish coloration will persist for several weeks. To detect nitrates, therefore, in the presence of nitrites it is necessary to compare a solution known to be free from nitrates with the one in question by the method given below under the "Determination of Nitrates."

The Presence of Nitrates in Milk.—Potassium nitrate has been used as a preservative, and also to remove the turnip-like taste. Samples of milk recently examined in this laboratory were found to contain potassium nitrate, the vendor stating in court that it had been added for this latter purpose. It is wise, therefore, to examine for nitrates as a routine test, and this is carried out conjointly with the estimation of fat by the Gerber method. When milk containing either nitrates or nitrites is shaken up with 90 per cent. sulphuric acid, a vivid brown colour is produced—golden-brown in the case of nitrites, somewhat darker in the presence of nitrates. This colour is very easily seen on shaking the milk in the Gerber tube with milks containing as little as 5 parts per 10^6. When such a colour is noticed, the milk should be further tested.

The Determination of Nitrates in Milk.—Tillmans (*loc. cit.*) recommends his diphenylamine method described above; but we have found that, although this gives good comparative results with such quantities of nitrate as are found in watered milks (1 per 10^6), the method is too delicate, however, for concentrations of nitrate about 2 per 10^5. As larger quantities than this have occurred in practice when potassium nitrate has been added to milk, a method was required for estimating them in such concentrations.

Sulphuric Acid Method.—Five c.c. of milk are placed in a stoppered cylinder, and 5 c.c. of sulphuric acid (sp. gr. 1·770) are added. The cylinder is then shaken, and the colour produced compared with that given with milks containing known quantities of nitrate treated in a similar manner. This method gives good results in the absence of nitrites; but as nitrates and nitrites are sometimes found together in milk, the following test, which is applicable in the presence of nitrites, is of more general application:

Brucine-Sulphuric Acid Method.—The reagent is prepared by dissolving 0·2 grm. of pure brucine in 75 c.c. of sulphuric acid (sp. gr. 1·82), and adding this to 25 c.c. of water.

In the absence of nitrites, 5 c.c. of the serum (*q.v.*) are placed in a test-tube, and 10 c.c. of the reagent are added. The contents are well mixed, poured into a Nessler cylinder and allowed to stand for half an hour. The colour is then compared with standards prepared in the same manner.

In the presence of nitrites the colour is produced immediately on the addition of the reagent, and then quickly commences to fade owing to the bleaching action of the nitrites. Such action is apparent even after standing all night, although the total effect is not great. We have found, however, that the bleaching effect due to the presence of nitrites upon the colour produced by nitrates is practically independent of the amount of nitrite present—at least, in quantities between 2 and 40 per 100,000.

In such cases of a mixture of nitrate and nitrite the procedure is as follows: Five c.c. of the serum are placed in a test-tube, and 10 c.c. of the reagent are added. At the same time standards are prepared containing suitable quantities of nitrate, and each containing about 10 per 100,000 of sodium nitrite. The contents of the tubes are poured into Nessler cylinders, and matched any time after three hours.

Reduction of Nitrates to Nitrites in Milk on Keeping.—A short time ago some samples of milk were examined in this laboratory, which, after standing, gave a faint nitrite reaction with the Griess-Ilosvay reagent, about 3 parts per 10^6 of sodium nitrite being found. The following day, repeating the determination, 10 parts per 10^6 were present. We eventually found this phenomenon to be due to the presence of nitrate in the milk, this nitrate being decomposed with the formation of nitrite. On going into the matter further, we found that if potassium nitrate be added to milk, after two days nitrite can be detected in the milk, and the quantity becomes appreciable after another day. In order to follow the course of this decomposition, two milks were prepared containing respectively 100 and 20 parts per 10^6 of potassium nitrate. These were tested for the presence of nitrite on five successive days, with the following results:

	Parts per 10^6 of Nitrite found.				
	1st Day.	2nd Day.	3rd Day.	4th Day.	5th Day.
Milk containing 100 per 10^6 KNO_3.	0	2	8	25	12
Milk containing 20 per 10^6 KNO_3.	0	0·2	2	4	0

It will be seen from these results that the proportion of nitrite formed increases to a maximum, and then decreases. The above figures were obtained starting with fresh milk, but similar figures were given by sour milk.

The Detection and Estimation of Nitrites in Milk.—The question of nitrites in milk came into prominence some little time ago in connection with a proprietary milk preservative called "Mystin," upon which a report was issued by G. W. Monier-Williams (*Food Reports*, Local Government Board, No. 17; ANALYST, 1912, **37**, 155); G. A. Stokes (*ibid.*, 1912, **37**, 178).

In both these papers it is stated that sodium nitrite interferes with the Hehner test for formaldehyde when present in the proportions found when using "Mystin." We have been unable to confirm these statements. With a Hehner solution, prepared by mixing 1,000 c.c. concentrated sulphuric acid and 25 c.c. of a 5·5 per cent. solution of ferric chloride, we obtained a distinct purple ring after four hours with a milk containing 1 part per 300,000 of formaldehyde and 1 part per 9,000 of sodium nitrite, which is the strength recommended if using "Mystin" as a preservative. The colour produced was certainly not as bright as in the absence of the nitrite, but it was unmistakable. We have repeated this on several occasions, and always with the same result.

G. A. Stokes (*loc. cit.*) suggests that the presence of the formaldehyde will be detected whilst performing the Gerber test, but this we are unable to do if nitrite be present. We find, however, that the presence of a nitrite is shown by the golden-brown colour produced on shaking the tube, the colour produced being somewhat brighter than that produced in the corresponding manner from a nitrate. This colour is unmistakable.

Estimation of Nitrites.—There are two methods in general use for the estimation of nitrites in small quantities—the metaphenylene-diamine and the Griess-Ilosvay methods. Of these the latter is by far the more delicate, and the colour produced is easier to match. The reagent is made up as follows: 0·1 grm. of α-naphthylamine is dissolved in 20 c.c. of glacial acetic acid, and mixed with 0·5 grm. of sulphanilic acid dissolved in 150 c.c. of warm 6 per cent. acetic acid, the whole being diluted to 300 c.c. with more 6 per cent. acid; 150 c.c. of 60 per cent. acetic acid are added to this. In carrying out the test, 5 c.c. of the milk are placed in a test-tube (4 by 0·5 in.), and 3 c.c. of the reagent are added. Milks containing known quantities of nitrite are treated in the same way. The colour produced is matched with the standards after about one hour. The reaction with sulphuric acid can also be made use of as a quantitative test. The procedure is identical to that given above for the estimation of nitrates.

The Detection and Estimation of Formaldehyde in the Presence of Oxidising Agents.—We have stated that nitrites only interfere slightly with the Hehner test for formaldehyde, and that it is possible to detect quantities greater than 1 in 500,000 even in the presence of quantities of nitrite, such as 1 in 5,000. Potassium nitrate seems to have no effect whatever on the Hehner test as carried out by us, and the same is true of potassium chlorate. In this respect our experience differs from that of Stokes and Monier-Williams (*loc. cit.*). For the estimation of formaldehyde in milk under ordinary circumstances we have found the Shrewsbury and Knapp method to be by far the most delicate and the most accurate of the methods yet suggested, but it breaks down in the presence of nitrite. A modification of this test, suggested by Monier-Williams (*loc. cit.*), in which the nitrite is destroyed by urea previous to the colorimetric estimation, has been found by us to work well, and to give results very near the truth. The presence of potassium nitrate does not affect the ordinary Shrewsbury and Knapp test.

The Preservative Action of Potassium Nitrate and Potassium Nitrite.—In the discussion following the paper by Stokes (*loc. cit.*) surprise was expressed that formaldehyde of a concentration of 1 in 300,000 was sufficient to preserve milk for three days, this opinion being based upon the statement by Stokes that "'Mystin' added to milk in the proportion of 3 c.c. per litre was found to be a fair preservative; thus, after three days, the acidity developed was only equivalent to 0·24 per cent. of lactic acid, whereas the same milk unpreserved showed an acidity equivalent to 0·69 per cent. of lactic acid." We have experimented with formaldehyde and "Mystin" as preservatives in these dilutions, and have found that practically the whole of the preservative action of "Mystin" is due to the sodium nitrite which it contains. In the following tables some of our results are given; the acidities are expressed as c.c. of $\frac{N}{10}$ alkali, using 10 c.c. of milk. The figures given are acidities so expressed:

Date.	Control.	With CH_2O 1 in 10^5.	With $NaNO_2$ 10 in 10^5.	With CH_2O 1 in 10^5 and $NaNO_2$ 10 in 10^5.
May 4, 1912 ...	1·8	1·8	1·8	1·8
May 6, 1912 ...	5·2	3·9	3·2	3·2

Date.	Control.	"Mystin" as recommended.	$NaNO_2$ 10 in 100,000.
May 9, 1912	1·9	1·9	1·9
May 10, 1912	4·2	2·3	2·3
May 11, 1912	7·8	3·3	3·3

Potassium nitrate has sometimes been used as a milk preservative, but its use has not become general on account of its inefficiency. We have done some work showing that it has, however, a slight preservative action. Thus a milk containing 10 parts of potassium nitrate per 100,000 in four days developed an acidity of 5·5, the same sample of milk to which 2 in 100,000 had been added showed an acidity of 5·9, whilst the untreated milk in the same time developed an acidity of 6·2.

In conclusion, we would express our indebtedness to Mr. J. F. Liverseege for his valuable criticism and advice.

City of Birmingham,
Analytical Department.

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NOTE ON COFFEE AND CHICORY.

By T. R. HODGSON, M.A., F.I.C.

A short time ago a vendor in the Midlands was summoned for selling a mixture of coffee and chicory not of the nature, substance and quality demanded; the Analyst's certificate stating that the sample "contained 10·1 per cent. of water," and in the Observations that the sample contained "4·1 per cent. excess of water. A well-roasted sample should not contain more than 6 per cent." With a view of testing this statement, a number of samples were purchased in the ordinary course of business in various parts of the country, and the percentage of moisture determined.

						Percentage of Moisture.
Coffee	100	per cent.				2·0
,,	,,	,,				4·9
,,	,,	,,				5·3
,,	,,	,,				5·4
,,	,,	,,				5·6
,,	,,	,,				6·0
,,	,,	,,				6·3
	57	,,	Chicory	43	per cent.	2·6
,,	94	,,	,,	6	,,	3·0
,,	66	,,	,,	34	,,	3·5
,,	67	,,	,,	33	,,	4·0
,,	70	,,	,,	30	,,	4·2
,,	44	,,	,,	56	,,	4·6
,,	70	,,	,,	30	,,	5·0
,,	50	,,	,,	50	,,	7·0
,,	87	,,	,,	13	,,	7·2
,,	56	,,	,,	44	,,	7·5
,,	50	,,	,,	50	,,	9·7
,,	44	,,	,,	56	,,	11·0
Chicory	100	,,				10·8
,,	,,	,,				11·3

Owing to the very large variation in the amount of moisture present, I thought it would be advisable to see whether a sample would absorb moisture on being kept under conditions similar to those under which it is usually kept in retail shops; one sample of pure coffee and one of pure chicory and one of a mixture of coffee and chicory were therefore placed in a drawer, such as is usually seen in small retail shops, and the moisture was determined at intervals.

"*Pure Chicory.*"

	Percentage of Moisture.
Original sample	11·3
After 3 days in the drawer	14·4
,, 7 ,, ,, ,,	15·7
,, 14 ,, ,, ,,	15·9
,, 21 ,, ,, ,,	15·9

"*Pure Coffee.*"

Original sample	2·0
After 3 days in the drawer	7·8
,, 7 ,, ,, ,,	9·5
,, 14 ,, ,, ,,	9·7
,, 21 ,, ,, ,,	9·7

"*Coffee* 66 *per Cent., Chicory* 34 *per Cent.*"

Original sample	3·5
After 3 days in the drawer	9·7
,, 7 ,, ,, ,,	11·2
,, 14 ,, ,, ,,	11·3
,, 21 ,, ,, ,,	11·5

It is obvious that coffee and chicory when kept absorb moisture somewhat rapidly, and therefore the standard of 6 per cent. of moisture which it was proposed to set up cannot stand.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Use of the Immersion Refractometer in Examining American Beers made from Malt and Unmalted Cereals. R. Schwartz. (*J. Ind. and Eng. Chem.*, 1913, **5**,660.)—Danzer (*Zeitsch. ges. Brauw.*, 1910, **33**, 32) has published a series of tables (based on the alcohol and extract tables of K. Windisch) from which the alcohol and extract of any beer can be calculated from the same determinations as those necessary when using Ackermann's tables (*ibid.*, 1905, **28**, 33). As there was some doubt whether Danzer's tables could be employed in the case of beers containing 60 to 70 per cent. of malt and 30 to 40 per cent. of adjuncts such as unmalted cereals, the author has examined with the aid of these tables and the refractometer forty-seven samples of beer, some of them being malt and hop beers only, some top fermentation beers, and the rest typical American substitute beers. The original gravity of the same beers by the official distillation and pycnometer method was done by another worker, and serves as the standard.

Differences in $\frac{1}{100}$ths per Cent.

		Plus.		Minus.	
	Average.	Highest.	Lowest.	Highest.	Lowest.
Alcohol ...	8	20	0	19	0
Extract	6	7	1	20	0
Original gravity ...	14	30	0	40	5

For calculating the original gravity, the formula $G = 1{\cdot}93\,A + E$ was used, and the results recorded to the nearest 0·05 per cent. The conclusion is reached that the agreement between the results of the two methods is as good for American beers as it is for European all-malt beers, and that the refractometer method may safely be used for brewery control and for all ordinary work, with the possible exception of legal and disputed cases. (*Cf.* ANALYST, 1908, **33**, 320.) H. F. E. H.

Determination of Small Amounts of Caffeine: A Comparison of Methods. B. L. Murray. (*J. Ind. and Eng. Chem.*, 1913, **5**, 668.)—Two methods are compared—that of Görter (ANALYST, 1908, **33**, 124) and that of Lendrich and Nottbohm (ANALYST, 1909, **34**, 214). The former gives results from 50 to 100 per cent. higher than the latter, especially when the total amount of caffeine present in the sample is 0·5 per cent. or less. The advantage of purifying the crude caffeine with permanganate as advised by Lendrich and Nottbohm is confirmed by experiments, and in all cases the caffeine percentages recorded were calculated from the nitrogen present, and not by weighing the caffeine itself. Duplicate analyses by different analysts using the Lendrich and Nottbohm method agree well. The author gives no indication as to which method he considers gives results nearest to the truth.

H. F. E. H.

Reactions of Digitoxine. C. Reichard. (*Pharm. Zentralhalle*, 1913, **54**, 687-693; through *Chem. Zentralbl.*, 1913, II., 719.)—Pure digitoxine crystallises in long needles, grouped in star formations. It is insoluble in water, but dissolves in chloroform and alcohol, and acetic acid, and is unattacked by cold sulphuric acid. Nitric acid (25 per cent.) produces no coloration, but potassium hydroxide gives a faint yellowish-red colour. The reactions of digitoxine with chromic acid or molybdic acid are very characteristic. When a trace of the alkaloid is rubbed with ammonium molybdate or potassium bichromate and water, the mixture allowed to evaporate, the residue moistened with acetic acid and again evaporated, and then treated with strong sulphuric acid, colourless solutions are obtained, and these on heating give pronounced colorations. The molybdate mixture gives a sky-blue colour and the chromium mixture a green colour, both of which disappear when the liquid cools, but reappear on heating. If a mixture of sodium metavanadate with solid digitoxine and water be dried, and the residue moistened with strong sulphuric acid, there is produced after a few hours a nearly colourless solution, which becomes deep yellow when gently warmed, and colourless again on cooling. Potassium ferrocyanide, applied in the same manner as described for molybdic acid, gives a deep blue coloration, changing to blackish-blue, whilst potassium ferricyanide gives a sky-blue coloration. Sodium nitroprusside, however, has no action upon digitoxine. Sodium tungstate gives an amorphous white precipitate, partially soluble in dilute sulphuric acid. Copper sulphate also yields an amorphous precipitate, which is darkened by sulphuric acid. Mercuric chloride, added to an acetic acid solution of digitoxine, on evaporation a residue is left which yields a blue solution when warmed with sulphuric acid, whilst mercuric nitrate gives a yellow liquid under the same conditions. Bromine-water has no action; hydrochloric acid gives a green coloration on warming, and ferric salts a deep red-brown colour. The greenish-yellow colour given by cobalt nitrate when evaporated at a gentle heat with digitoxine and acetic acid is characteristic.

C. A. M.

Estimation of Nitrobenzene in Earthnut Oil. H. J. Lucas. (*J. Ind. and Eng. Chem.*, 1913, **5**, 576-577.)—The author has found the following process to afford a satisfactory means of estimating nitrobenzene in earthnut oil, oil thus flavoured being largely consumed by the poorer classes in Porto Rico. The process consists, essentially, in reducing the nitrobenzene to aniline by means of zinc and hydrochloric acid, extracting the aniline, and weighing it as aniline hydrochloride; about 95 per cent. of the nitrobenzene present is recovered. The oil is shaken with zinc dust and hydrochloric acid for fifteen minutes, and then with a mixture of ether and water. After several shakings with this mixture, the separated aqueous layers are filtered, mixed with sodium hydroxide solution, and extracted with ether. The ethereal extracts are now shaken with hydrochloric acid, and the acid extractions are evaporated in a tared platinum basin. The evaporation is stopped before complete dryness is reached, and the moist residue is dried over soda-lime for from sixteen to thirty-five hours. The dry residue of aniline hydrochloride is then weighed and afterwards ignited, any residue of inorganic salts thus obtained being deducted from the first weight. W. P. S.

Viscosity of Butter Fat and its Substitutes. G. F. White and R. H. Twining. (*J. Ind. and Eng. Chem.*, 1913, **5**, 568-573.)—It is suggested that determinations of the viscosity of butter fats may afford a means of detecting the presence of margarine. Margarine fats are always more viscous than butter fat, except when the former contains a large proportion of oils. Results of numerous determinations of the viscosities of butter fats, margarines, and mixtures of the same are recorded, the apparatus employed for the determinations being a modification of that described previously by one of the authors (ANALYST, 1912, **37**, 143). W. P. S.

Differentiation of Animal and Vegetable Fats. J. Marcusson and H. Schilling. (*Chem. Zeit.*, 1913, **37**, 1001-1002.)—The method described is a combination of Bömer's phytosteryl acetate test with Windaus' digitonine precipitation of cholesterol or phytosterol (ANALYST, 1910, **35**, 256). No preliminary saponification is required, the fat being merely shaken with a dilute alcoholic solution of digitonine: Fifty grms. of the sample are shaken for fifteen minutes with 20 c.c. of a hot 1 per cent. solution of digitonine in 96 per cent. alcohol, and allowed to stand for several hours. The oil is drawn off as completely as possible, and the separated digitonides in the upper alcoholic layer are shaken with 50 to 100 c.c. of ether, then filtered, and washed with ether until free from oil. They are next heated for thirty minutes in a test-tube with 1·5 c.c. of acetic anhydride, and the acetates which separate upon cooling are recrystallised and examined in the usual way. The method gives good results in the separation of the alcohols from blown and oxidised oils, and the presence of paraffin wax or mineral oils does not interfere with the test. By means of this method a small quantity of cholesterol was isolated from sperm oil. The acetates from a mixture of 90 per cent. of sperm oil and 10 per cent. of rape oil melted after two recrystallisations at 116° to 117° C., whilst when 5 per cent. of rape oil was present the melting-point of the acetates was 114° to 116° C. C. A. M.

Fatty Acids of Kaya Oil. S. Ueno. (*Chem. Rev. Fett Ind.*, 1913, **20**, 208-209.)—The fatty acids of the oil examined by the author had a mean molecular weight of 286·2, and an iodine value of 139·9. Examined by the usual methods, they were found to consist of about 9 per cent. of palmitic and stearic acids, 19 per cent. of oleic acid, and 72 per cent. of a linolic acid which yielded a sativic acid melting at 151° to 152° C. (instead of the usual m.-pt. 174° C.). C. A. M.

Detection of Paraffins in Lanolin. G. Tellera. (*Boll. Chim. Farm.*, 1913, **52**, 1-4; through *Chem. Zentralbl.*, 1913, II., 818.)—The following test is proposed for the detection of vaseline in lanolin: One grm. of the sample is dissolved in ether, and the stearin which separates on cooling the mixture is removed by filtration. Five c.c. of absolute alcohol are then added to the filtrate. When the lanolin contains from 3 to 4 per cent. of vaseline, a flocculent precipitate is obtained immediately, whilst when only 1 per cent. of vaseline is present the precipitate appears after the lapse of about thirty minutes. Crude lanolin gives a slight turbidity on the addition of the alcohol, but purified lanolin gives a clear solution. W. P. S.

Constituents of Hops. F. B. Power, F. Tutin, and H. Rogerson. (*J. Chem. Soc.*, 1913, **103**, 1267-1292.)—Fresh air-dried Kentish hops (1911) containing 10·5 per cent. of moisture were used for this investigation, and indications were obtained of the presence of a very small amount of an alkaloid. The tannin as estimated by Chapman's cinchonine method (ANALYST, 1908, **33**, 95, and 1909, **34**, 372) was 2·40 per cent. Twenty-five kgrms. of the hops were completely extracted with hot alcohol, and the extract distilled in a current of steam to remove the essential oil. From the portion of the extract soluble in water were isolated small amounts of choline and *l*-asparagine. The aqueous liquid also contained in addition to tannin a quantity of potassium nitrate, and a sugar yielding *d*-phenylglucosazone as well as an amorphous bitter-tasting substance. The portion of the alcoholic extract insoluble in water consisted of a dark green oily resin, equivalent to 14 per cent. by weight of the hops, and from this were isolated ceryl alcohol, hentriacontane ($C_{31}H_{64}$), a phytosterol, a phytosterol glucoside, a mixture of volatile fatty acids (formic, acetic, butyric, etc.), β-isopropylacrylic acid, saturated and unsaturated non-volatile acids (palmitic, stearic, cerotic, and an isomeride of arachidic acid); chrytinic acid, binolic acid, a new crystalline phenolic substance, humulol, having a pale fawn colour and a bitter taste, which on hydrolysis with caustic potash yielded, amongst other products, an acid ($C_{15}H_{14}O_5$, m.-pt. 210°), and *p*-hydroxybenzaldehyde; a new crystalline phenolic substance, xanthohumol ($C_{13}H_{14}O_3$, m.-pt. 172°), which is tasteless and has an orange-yellow colour. The presence of a yellow colouring matter resembling quercitrin or of a wax consisting of myricyl palmitate was not confirmed. The bitterness of hops is not due to any single substance, such as "hop-bitter acid" or "lupulic acid," but is to be attributed to a number of products mostly amorphous. The differentiation of the resinous material as α-, β-, and γ-resins is misleading, since it is much more complex than would be indicated by the methods used for their separation. As the resinous material contains a large proportion of the ordinary fatty acids and their esters, such methods for the valuation of hops as are based on the titration of extracts obtained by means of light petroleum, etc., are of very doubtful utility. H. F. E. H.

Nitrogenous Constituents of Hops. A. C. Chapman. (*Chem. Soc. Proc.*, 1913, **29**, 182-184.)—The material employed was either hops extracted in the laboratory or hop-extract prepared on the commercial scale. Four different methods of preparing and separating the hop constituents were employed. By the first method the concentrated aqueous extract was precipitated with basic lead acetate, and the filtrate, after removing the excess of lead, was precipitated with phosphotungstic acid. Histidine, betaine, and choline were thus isolated, and, from the phosphotungstic filtrate, asparagine. In the second method the hops were mixed with lime, boiled with water, and the mixture evaporated, dried, and extracted with alcohol, the extract freed from alcohol being then precipitated with phosphotungstic acid. The solution of the bases so obtained, on precipitation with an ammoniacal solution of silver chloride, yielded adenine and hypoxanthine. In the filtrate from the purine precipitate betaine and choline were again obtained. In the third method the hops were extracted with ammoniacal amyl alcohol, which was then treated with dilute hydrochloric acid. On treatment with phosphotungstic acid, adenine, betaine, choline, and a trace of some alkaloid, were obtained. Further experiments were carried out upon a concentrated aqueous extract of hops, which was treated with a mixture of alcohol and acetic acid, filtered, and the filtrate freed from alcohol, the residue being extracted with water. Amino-acids, (?) polypeptides and potassium nitrate were among the substances so obtained. H. F. E. H.

Extension of Vieth's Table for Correcting the Specific Gravities of Milk. C. H. Wright. (*J. Soc. Chem. Ind.*, 1913, **32**, 777-778.)—For the convenience of workers in tropical climates, the author has extended Vieth's correction table so that it may be used in the case of specific gravities determined at temperatures between 24° and 31° C. The table is used in the same way as Vieth's; if, for example, the sp. gr. of a milk is 1·028 at 28° C., its sp. gr. at 15·6° (60° F.) is 1·0311.

Degrees of Lactometer.	Temperature in Degrees Centigrade							
	24.	25.	26.	27.	28.	29.	30.	31.
20	21·7	21·9	22·1	22·4	22·6	22·8	23·1	23·3
21	22·7	23·0	23·2	23·4	23·7	24·0	24·2	24·5
22	23·7	23·9	24·2	24·4	24·7	25·0	25·2	25·5
23	24·7	24·9	25·2	25·4	25·7	26·0	26·2	26·5
24	25·7	26·0	26·2	26·5	26·8	27·0	27·3	27·6
25	26·8	27·0	27·3	27·5	27·8	28·1	28·3	28·6
26	27·8	28·1	28·4	28·6	28·9	29·2	29·5	29·8
27	28·9	29·2	29·4	29·7	30·0	30·3	30·5	30·8
28	30·0	30·2	30·5	30·8	31·1	31·4	31·7	32·0
29	31·0	31·3	31·6	31·9	32·2	32·5	32·8	33·1
30	32·1	32·4	32·7	33·0	33·3	33·6	33·9	34·2
31	33·1	33·4	33·7	34·0	34·4	34·7	35·0	35·4
32	34·3	34·6	34·9	35·2	35·6	35·9	36·2	36·6

W. P. S.

Morres's "Alizarol" Test for Milk. A. Devarda and A. Weich. (*Arch. Chem. u. Mikroskopie*, 1913, 1-6; through *Chem. Zentralbl.*, 1913, II., 718.)—The acidity of milk may be estimated with sufficient accuracy for practical purposes by the colour obtained in the alizarol test (Analyst, 1912, **37**, 26), but not when the acidity is pronounced or alkaline decomposition products have been produced.

When the coagulation caused by the alizarol test does not correspond to the acidity, abnormal composition, or decomposition of the milk due to coagulating and peptonising bacteria, is indicated. C. A. M.

Determination of Sodium Borate in Soap. P. Poetschke. (*J. Ind. and Eng. Chem.*, 1913, **5**, 645.)—The usual method of extracting soap with absolute alcohol, and estimating the borax in the insoluble residue by titration, is inaccurate, owing to the solubility of borax in alcohol, an error of about 20 per cent. being thus introduced. Attempts to determine the borax by ignition of the soap with alkali carbonates and titration in presence of glycerol failed owing to the presence of silica. The following method, adapted from Wherry and Chapin (ANALYST, 1909, **34**, 34) was employed successfully: Ten grms. of the soap (or 5 grms. if more than 5 per cent. of borax is present) are weighed into a platinum dish, and 2·15 grms. of fusion mixture, consisting of 200 grms. of sodium carbonate and 15 grms. of silica in fine powder, are added, together with 15 c.c. of alcohol. The whole is evaporated to dryness on the water-bath, and then ignited and fused, after which the mass is boiled out with water, transferred to a 250 c.c. round-bottomed flask, and acidified with 20 c.c. of dilute (1 : 1) hydrochloric acid. The solution is heated nearly to boiling, and then a slight excess of precipitated calcium carbonate is added, after which the whole is boiled for ten minutes under a reflux condenser. The insoluble matter is filtered off and washed, and the filtrate returned to the flask with a small quantity of calcium carbonate, and again boiled under the reflux condenser, then cooled, and titrated in presence of 50 c.c. of neutral glycerol with $\frac{N}{10}$ sodium hydroxide, using phenolphthaleïn as indicator. Figures are given showing that known amounts of borax added to borax-free Castile soap can be accurately determined. Many "borax soaps" are free from any borax, others contain from 1 to 10 per cent.

H. F. E. H.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Estimation of Chlorine in Blood. H. Rogée and C. Fritsch. (*Biochem. Zeitsch.*, 1913, **54**, 53-59.)—The method differs from that of K. O. Larsson (ANALYST, 1913, 270) in that the proteins are removed by iron according to Michaelis and Rona, and a correction is applied for the chlorine contained in the iron solution. The blood (10 c.c.) is weighed into a 250 c.c. flask, diluted with 150 c.c. water, 50 c.c. *liquor ferri oxydati dialysiti* added, and, after adding 5 c.c. of a 20 per cent. magnesium sulphate solution, the liquid is made up to the mark with water and shaken vigorously for some seconds. After allowing to settle and filtering, the chlorine in the filtrate is estimated according to Mohr's method. A blank experiment without blood is carried out in order to apply the necessary correction. The method can be suitably modified for micro-estimations. E. W.

Disinfectant Value of Mercuric Hydroxycyanide. H. Kühl. (*Arch. Pharm.*, 1913, **251**, 340-349.)—When added in very dilute solution 1 : 200,000 to culture media, mercuric hydroxycyanide stimulated the growth of bacteria, but in stronger concentrations inhibited and destroyed bacterial growth. For example, 1 mgrm. of the hydroxycyanide added to 25 c.c. of raw milk had a strong restrictive effect upon the growth of bacteria, 3 mgrms. destroyed streptococci after three days, whilst 5 mgrms. killed all micro-organisms. The same results were also obtained with mercuric cyanide containing only 33 per cent. of hydroxycyanide. In the author's opinion, although the OH ions play a considerable part in the disinfecting process, the relationship between the cell protoplasm and the poison is of much more importance. The fact that in certain experiments in which common salt was also used there was a more pronounced germicidal effect, is attributed not to the direct action of the sodium chloride, but to its promoting the absorption of the poison by the protoplasm. C. A. M.

Physiological Standardisation of Digitalis Preparations. R. Heinz. (*Merck's Jahresber.*, 1913, **26**, 1-20; through *Chem. Zentralbl.*, 1913, II., 621.)—The method depends upon an estimation of the minimum dose required to arrest the heart's action in twelve hours when subcutaneously injected into a frog, the results being compared with those obtained with a standard preparation of strophanthine of known physiological action. Taking the value of the strophanthine as 1,000, the following results were obtained: Digitoxine, 200; digitaline, 100; and powdered digitalis leaves, 0·5. Comparative experiments have also been made upon rabbits, cats, and mice. C. A. M.

Biochemical Examination of Soil. J. Stocklasa. (*Chem. Zeit.*, 1913, **37**, 1005.)—Reference is made to the author's method of measuring the respiration intensity of the bacteria in soil, and of the measurement of the quantity of nitrogen consumed by soil bacteria. When oxygen is excluded, decomposition of nitrogenous organic substances takes place, with putrefactive changes. These decompositions, and also the oxidation changes produced by aerobic bacteria, may be estimated by biochemical methods. Estimations of the capacity of the soil for decomposing cellulose, of the catalase in the soil, and of the biological absorption, ought also to be made. C. A. M.

Significance of the Estimation of the Reaction and Basicity of Soil. H. R. Christensen. (*Chem. Zeit.*, 1913, **37**, 1005.)—Estimation of the reaction of the soil affords an index, not only of the need of lime, but also of other substances required by plant life. It may also show whether the soil can offer resistance to certain plant diseases. The biological method will indicate, not only the lack of lime, but also whether an addition of other nutrient substances is required. Results so far obtained tend to show that the introduction of nitrogen in the form of nitrates gives better results than when the nitrogen is added to the soil in the form of cyanamide or ammonium sulphate. As regards potassium fertilisers, it appears that the injurious effects sometimes observed when chlorides are present in the potassium salts are less pronounced when the soil is acid than when it is alkaline. C. A. M.

Sources of Error in the Estimation of Sugar in the Blood of Frog and Tortoise. E. J. Lesser. (*Biochem. Zeitsch.*, 1913, **54**, 252-256.)—The filtrate obtained after removing proteins by means of colloidal iron from the blood of frogs or tortoises, contains substances which dissolve cuprous oxide, and therefore interfere with sugar estimation. They can be removed, however, by precipitation with mercuric nitrate, and the filtrate, after being freed from mercury with sulphuretted hydrogen, is treated with sodium acetate, and after concentration on the water-bath can be used for the sugar estimation. E. W.

Estimation of Small Amounts of Glucose in Urine. E. Hirschberg. (*Zeitsch. Physiol. Chem.*, 1913, **86**, 484-494.)—Bertrand's method, modified as suggested by Oppler (ANALYST, 1912, **37**, 140), by adding alcohol in order to lower the boiling-point, yields reliable results for glucose contents ranging from 0·50 to 0·90 per cent. For smaller glucose contents up to 0·10 per cent. the method is not so reliable, but frequently gives good results. The separation of cuprous oxide is incomplete in too dilute solutions. Bang's method (ANALYST, 1910, **35**, 70) gives results which are consistently high, and is useless for sugar contents of less than 0·40 per cent. E. W.

Detection of Urobilin. T. Hausmann. (*Zeitsch. exper. Path. Ther.*, 1913, **13**, 373-399; through *Chem. Zentralbl.*, 1913, II., 819.)—The method depends on the fact that the urobilinogen of urine is oxidised by copper sulphate to urobilin, which may be extracted with chloroform. Twenty c.c. of the urine are mixed with 2 c.c. of a 10 per cent. copper sulphate solution, and the mixture is shaken with 2 c.c. of chloroform. The chloroform layer is coloured pink, orange, or red; in the case of very acid urines the colour obtained is yellow. If, in place of copper sulphate, the urine is treated with a concentrated solution of either ferrous sulphate, zinc acetate, zinc sulphate, or potassium ferricyanide, the chloroform layer remains colourless, whilst lead acetate, mercuric chloride, and phosphotungstic acid yield precipitates with the urine, and only a small proportion of the urobilin is extracted by the chloroform. Previous treatment of the urine with formaldehyde or dimethylaminobenzaldehyde hinders the oxidation of the urobilinogen. The chloroform extract contains the actual urobilin and not its copper compound; the urobilin may be extracted from its chloroform solution by shaking the latter with sodium hydroxide solution; the alkaline solution exhibits the characteristic properties of a urobilin solution (absorption bands, fluorescence with alcoholic zinc acetate, etc.). For the estimation of the urobilin, the urine is treated with copper sulphate and extracted many times with chloroform; the chloroform extracts are evaporated to dryness, the dry residue is dissolved in $\frac{N}{10}$ sodium hydroxide solution, and the excess of the latter is titrated with $\frac{N}{10}$ hydrochloric acid; the difference between the quantities of alkali and acid used is multiplied by 0·0062 to give urobilin. W. P. S.

ORGANIC ANALYSIS.

Estimation of Petroleum Spirit in Turpentine. C. Bakker. (*Chem. Weekblad*, 1913, **10**, 420; through *Chem. Zentralbl.*, 1913, II., 179.)—To 10 to 25 c.c. of the turpentine is added, with cooling, three times its volume of strong sulphuric acid; the liquid is allowed to stand for twenty-four hours, and the proportion not attacked by the acid determined. This is then allowed to drop into three times its volume of fuming nitric acid, with cooling, and the unattacked layer is pipetted off, washed with water until neutral, distilled with steam, and the volume of petroleum spirit passing over read off. O. E. M.

Estimation of Brisance and of Velocity of Detonation. H. Kast. (*Zeit. Schiess- u. Sprengstoffwesen*, 1913, **8**, 65, 88, 133, 155, 172.)—The brisance of an explosive may be expressed approximately as $B=\frac{E}{t}d$, or $B=E\frac{V}{l}d$, in which E is the quantity of energy set free in time t, d is the gravimetric density of the explosive, and represents the energy concentration, V the velocity of detonation, and l the length of explosive over which the measurement is made. The direct determination of brisance is not possible; the lead block gives figures which are merely comparative. The velocity of detonation has been measured in various ways; the Siemens spark chronograph is the most accurate (*cf.* Kast, "Anleitung," p. 1025), and was used in this investigation; its chief sources of error are considered. The velocity of detonation increases as the diameter, strength of enclosure, and density of the explosive are increased. The strength of enclosure has no material influence, however, except at low densities of loading. With nitro-compounds, such as trinitrotoluene, picric acid, etc., the limiting diameter was 10 mm., no increase beyond this, up to 300 mm., which was the largest diameter tried, causing a higher rate of detonation; the statement that the propagation of a detonative wave takes place only in narrow tubes is not supported. With ammonium nitrate explosive of low density the velocity increased up to a limiting diameter of 40 to 50 mm. The observation of Dautriche, that there is a limiting density beyond which the velocity falls off, was not confirmed in the case of nitro-compounds, no such limit being reached with the densities employed; but with a mixture such as monachit the limit was found to be 1·2, beyond which the velocity fell off. The tendency to incomplete detonation, shown by a diminishing velocity in successive portions of the length tested, and most marked with plastic explosives, is due to an insufficiently powerful detonator. On the other hand, cases occur in which the velocity rises, as the detonative wave advances, to the normal rate. In the use of explosives, if their full energy is to be developed, the density and means of ignition must be such that the normal rate of detonation is reached; this is ascertained by a determination of the velocity of detonation. This principle may also be applied to the testing of explosives, such as dynamite and cheddite, which on storage become more difficult to detonate, and hence lose power unless larger detonators are used. The influence of chemical composition, which with nitro-compounds is much less important than the density, and of other factors, is considered. O. E. M.

Simple Method for the Determination of Carbon in Organic Materials. E. B. Hart and K. J. Woo. (*J. Amer. Chem. Soc.*, 1913, **35**, 1056.)—The principle of the method is the fusion of the material with sodium peroxide in an ordinary crucible, whereby all of the carbon is converted into sodium carbonate. This is distilled with 25 per cent. sulphuric acid, the escaping carbonic acid being collected in caustic soda and titrated by the double titration method, using phenolphthaleïn and methyl orange as indicators. The method gives accurate results and is specially suitable for routine carbon determinations in soils, and also where it is desired to follow this element in certain phases of animal nutrition, as in urine analysis. Materials low in carbon content prove most satisfactory. About 6 grms. of the peroxide and from 0·1 to 2 grms. of the material are ignited in an enclosed crucible of porcelain, nickel or iron, which must be of smooth interior surface. For soils 1 to 2 grms. are employed unless peaty, when as little as 0·1 to 0·2 grm. is taken. The burner should be withdrawn at the first signs of action, or the reaction may proceed with too great violence. The whole determination can be completed in three-quarters of an hour. H. F. E. H.

Studies on Caoutchouc Resins. M. Klassert. (*Zeitsch. angew. Chem.*, 1913, **26**, 471-472.)—The resins obtained in the purification of caoutchouc are now prepared on the commercial scale. The following samples have been examined by the author—No. 1: The crude resin, yellowish, opaque, with conchoidal fracture, and an odour of caoutchouc even in the cold. No. 2: The same, dried and fused, yellowish-brown, transparent, with a fracture like colophony, becoming strongly electrified when powdered, odourless in the cold, but developing an odour when rubbed or heated. No. 3: The same as No. 2, powdered, a yellowish powder. No. 4: The crude resin specially purified, a nearly white powder, practically odourless in the cold. No. 5: A greenish-brown, pasty mass, with microscopic crystals, apparently containing chlorophyll, and probably isolated by means of volatile solvents. For the sake of comparison some results, published by Vaubel and Diller (*Gummi Zeit.*, 1912, 1587), are also given in the table:

	No. 1.	No. 2.	No. 3.	No. 4.	Vaubel and Diller.	Fluid Resin. No. 5.
Moisture	3·6	0·2	0·3	25·10	—	8·4
Ash	1·0	2·5	1·1	0·8	0·5–2·1	0·3
Melting-point °C. ...	112°–118°	92°–98°	110°–118°	114°–119°	90°–125°	—
Hübl iodine value ...	34·2	36·3	36·0	28·3	20–41	82·3, 67·1
Saponification value ...	90·5	85·9	86·8	62·3	19–24	77·5
Acid value	1·3	1·2	1·3	0·9	3·2–13·6	23·8
Ester value (by difference)	89·2	84·7	85·5	61·4	—	53·7

The melting-points were extremely deficient in sharpness, and the samples were not really liquid until above 130° C. In the case of No. 5, the iodine value decreased considerably after heating the sample at 110° C. to expel volatile matters.

The solid resins are soluble in most organic solvents when heated, but separate for the most part on cooling, the acid constituents, however, being more soluble than the rest. Mixtures of equal parts of resin and oils, including paraffin oil, give plastic pastes, which are less "greasy" than pastes similarly prepared with colophony. Solutions of the resin in chlorinated hydrocarbon solvents show no separation of the constituents, and such solutions are suitable for lacquers. A number of products, possibly of technical interest, are obtained by destructive distillation of the resin: Total distillate, 85 per cent.; pitchy residue, 8 per cent. J. F. B.

Estimation of Cholesterol Bodies in Presence of Each Other. J. Lifschutz. (*Biochem. Zeitsch.*, 1913, **54**, 212-236.)—The spectral intensities of the acetic anhydride sulphuric acid reaction for cholesterol bodies, and the acetic acid-sulphuric acid reaction for oxycholesterol, were found to be proportional to the amounts of these substances in the respective reaction mixtures. The spectral phenomena of the acetic anhydride sulphuric acid reaction of such cholesterol bodies as give this reaction were found to be fundamentally identical. The method is based upon these two facts. The total cholesterol bodies are determined spectrometrically by means of the acetic anhydride sulphuric acid reaction carried out in chloroform solution and the oxycholesterol determined by the acetic acid sulphuric acid reaction as described (ANALYST, 1913, 209). It has been found that the acetic anhydride sulphuric acid reaction in presence of chloroform indicates the total cholesterol, whilst, in the absence of chloroform, free cholesterol, but not that in the form of fatty ester, is indicated. E. W.

Detection of Formaldehyde with p-Nitrophenylhydrazine. E. Zerner. (*Monatsh. für Chem.*, 1913, **34**, 957-961.)—In studying Bamberger's reaction for the detection of small quantities of formaldehyde by means of *p*-nitrophenylhydrazine, the author obtained a sparingly soluble body other than the normal hydrazone described by Bamberger, and melting at a higher temperature. The new body need not interfere with the performance of the test in the ordinary way, since it is formed only when a considerable excess of formaldehyde is present. Under normal conditions, with excess of *p*-nitrophenylhydrazine, Bamberger's hydrazone, melting at 181° C. after crystallisation from benzene, is obtained. For the preparation of the new body, 1 grm. of *p*-nitrophenylhydrazine hydrochloride is dissolved in 10 c.c. of boiling water, the solution is filtered, and, while at a temperature of about 65° C., is poured quickly into 5 c.c. of cold 40 per cent. formaldehyde solution. A precipitate of small yellow crystals is formed, which, after separation, washing, and recrystallisation from benzene, melt somewhat indefinitely between 222° and 225° C. Analysis of the product indicates a formula of $C_{14}H_{11}N_5O_4$, which would correspond with a condensation product formed from 2 molecules of formaldehydenitrophenylhydrazone with the loss of 1 molecule of ammonia. Such a body might have the constitution of a 1, 4-bisnitrophenyl-1, 2, 4-dihydrotriazol, and the substance obtained does not show the coloration with alcoholic potassium hydroxide characteristic of the imino-group in ordinary nitrophenylhydrazones. J. F. B.

Colour Reaction for Gallic Acid and Tannin. O. Schewket. (*Biochem. Zeitsch.*, 1913, **52**, 271-275).—A solution of gallic or tannic acid is treated with an excess of a 1 per cent. solution of iodine in potassium iodide, shaken, and tap-water or a very weak solution of a salt with alkaline reaction added, when a reddish-violet coloration is produced. Weak organic acids and hydriodic acid do not interfere, but even dilute mineral acids prevent the reaction. Concentrated alkaline hydroxides and carbonates disturb the reaction and give a dirty brown coloration. The reaction can be employed to distinguish gallic and gallo-tannic acids from tannins which are free from gallic acid. E. W.

Estimation of Free Sulphur in Leather. Jalade. (*Ann. Falsific.*, 1913, **6**, 435-446.)—When hides are treated with sulphur in the tanning process, the resulting leather frequently contains a quantity of free sulphur; as a rule, carefully manufactured leather does not contain more free sulphur than corresponds with 0·36 per cent. of SO_3, and any leather which contains more than this quantity (calculated on the dry material) is considered to be of unsatisfactory quality, as the sulphur oxidises gradually to sulphuric acid, and the strength of the leather is thus destroyed. The quantity of free sulphur is calculated from the difference between the quantity of sulphate in the ash of the leather and the amount of sulphate found in the ash after the leather has been treated with potassium carbonate (Balland and Maljean's method). In order to obtain concordant results, it is essential that a sufficient quantity of potassium carbonate be used, and that the leather be in a suitable state of division. The following procedure is recommended: The leather is cut up into thin shavings by means of a plane (Stanley's No. 220), and 5 grms. of the dried shavings are mixed in a crucible with 40 c.c. of a 1 per cent. potassium carbonate solution; the mixture is evaporated on a water-bath, dried for a short time at 110° C., and then ignited for about thirty minutes in a muffle-furnace. The crucible should be placed near the mouth of the muffle. When cold, the contents of the crucible are moistened with hydrochloric acid, evaporated to separate the silica, and the sulphate is then estimated in the usual way by precipitation with barium chloride. W. P. S.

Source of Error in the Kjeldahl-Gunning Method. E. Carpiaux. (*Ann. Chim. anal.*, 1913, **18**, 315-316.)—The fact that loss of ammonia takes place in this method when the composition of the acid digestion mixture approximates to that of potassium hydrogen sulphate, as mentioned by Self (ANALYST, 1912, **37**, 203), is fully confirmed by a series of experiments carried out by the author. To avoid such loss of ammonia, it is recommended that only moderate quantities of potassium sulphate should be employed, and that the heating should not be unduly prolonged after the digestion is completed. W. P. S.

Kapok Seeds and Kapok Oil. H. Sprinkmeyer and A. Diedrichs. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1913, **26**, 86-101.)—Kapok seeds are obtained from *Eriodendron anfractuosum*, a plant growing in almost all tropical countries. The plant resembles the cotton-plant in that the seeds are embedded in a mass of fibrous material which may be used for textile purposes. The seeds obtained from *Bombax malabaricum*, are also known as kapok seeds, and in commerce little, if any, distinction is made between the two kinds of seeds. About 18 per cent. of oil is obtained from the seeds by hot pressing, the actual oil content of the seeds varying from 19 to 26 per cent. The expressed oil varies in colour from yellow to dark brown, and sometimes deposits a mass of solid glycerides. The following results were obtained on the examination of expressed oils obtained from kapok seeds from Java, German East Africa, Ceylon, and Ecuador: Sp. gr. at 15° C., 0·9235 to 0·9326; refractometer reading at 40° C., 51·7 to 59·7; iodine value, 85·2 to 93·5; acid value, 18·5 to 210·2 (corresponding with from 5·22 to 59·3 per cent. of free oleic acid); saponification value, 189·2 to 194·5. The fatty acids separated from the oils had: Iodine value, 88·7 to 98·9; mean molecular weight, 277·6 to 282·1; m.-pt. 32·2° to 34·2° C.; solidifying-point, 26·9° to 31·8°. The expressed oil from the seeds of *Bombax malabaricum* gave the following results: Sp. gr. at 15° C., 0·9300; refractometer reading at 40° C., 57·0; iodine value, 73·6; acid value, 3·0 (equivalent to 0·85 per cent. of oleic acid); saponification value, 194·3. Kapok oil yields a more intense coloration with Halphen's reagent than does cottonseed oil; but Milliau's test (ANALYST, 1905, **30**, 98) serves to distinguish these two oils from one another. (See also ANALYST, 1903, **28**, 40, 320). W. P. S.

Drying Rates of Raw Paint Oils. L. V. Redman, A. J. Weith, and F. P. Brock. (*J. Ind. and Eng. Chem.*, 1913, **5**, 630-636.)—The object of this research was to compare the relative drying qualities of a series of raw oils without the use of dryers. Further work with standard dryers is in progress. The method adopted was the weighing at daily intervals of a series of films spread on glass plates which were exposed to a brisk current of air and but poorly illuminated by diffused daylight. Elaborate precautions were taken to secure uniform conditions and an abundant supply of dust-free air. The apparatus employed is figured in detail in the paper. The air passed over the films with a speed of about ten miles per hour, and in each second about four times as much oxygen passed through the apparatus as was actually absorbed by the oil films in twenty days. Chinese wood oil attained its maximum weight on the ninth day, whereas linseed oil under the same conditions reached its slightly greater maximum two days earlier. The Chinese wood oil set sooner and gave the harder film, but this had a frosted appearance. Soya oil reached its maximum weight on the sixth day and yielded a transparent film, but one much less tough than that given by linseed oil. The maximum increase in weight was under 8 per cent., against the 12 per cent. of linseed oil and 10·5 per cent. of Chinese wood oil. Fish oils of several kinds were experimented with, and showed very rapid absorption of oxygen, the maximum increase—about 13 per cent.—being reached in less than four days, but in no case was a solid film obtained in twenty days. G. C. J.

Examination of Steam Turbine Oils. F. Schwarz and J. Marcusson. (*Chem. Rev. Fett Ind.*, 1913, **20**, 215-220.)—The following modification of Kissling's method of estimating the tar-forming capacity of oils is recommended: Fifty grms.

of the sample are heated for fifty hours in an Erlenmeyer flask (lower diameter 7·5 cm., height 12·5 cm.) in a large oven, the temperature of which is kept constant at 120° C. The oil is then mixed with 50 c.c. of 50 per cent. (by weight) alcohol and 50 c.c. of 4 per cent. sodium hydroxide solution, and heated for fifteen minutes at about 80° C. beneath a reflux condenser, after which it is vigorously shaken for five minutes and allowed to stand overnight in a separating funnel. The alkaline layer is then drawn off as completely as possible, and extracted with 30 c.c. of petroleum spirit to remove any oil. It is next acidified with hydrochloric acid, and shaken with three successive portions of 50 c.c. each of benzene, the united benzene extracts washed with water and evaporated, and the residue of tar dried for ten minutes at 150° C., and weighed and calculated upon 50 c.c. (not 50 grms.) of the oil. A further point to be observed in the selection of turbine oils is that they should not readily emulsify with water, since in the presence of water and metal the resinification capacity is increased. From the examination of a large number of samples the authors conclude that the most suitable oils for turbines are those with a viscosity of 9° to 14° Engler at 20° C.; sp. gr. of 0·870 to 0·905 at 15° C.; and a tar-forming value not exceeding 0·2. The oils should be pure mineral oils free from acid and soaps, and should not contain more than 0·01 per cent. of ash. The resinification of mineral oils appears to be due to the presence of resinous viscous substances of high sp. gr. and optical refraction. As a rule they contain sulphur and oxygen, and are apparently derived from unsaturated terpenes. C. A. M.

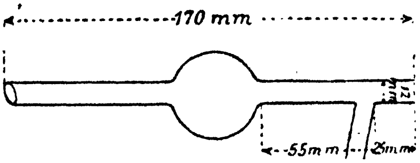
Fractionation of California Petroleum by Diffusion through Fuller's Earth. J. E. Gilpin and P. Schneeberger. (*Amer. Chem. J.*, 1913, **50**, 59.)—The investigation of the effect produced on petroleums by diffusion through fuller's earth was extended to a very heavy petroleum from California. It was found that for petroleums of sp. gr. 0·912 at 20° C. there was no fractionation unless the oil was heated to 75° C.; from petroleums containing sulphur compounds light fractions were separated, containing less sulphur than the original oil. Nitrogen compounds were removed by upward diffusion of an oil. Mixtures of benzene and paraffin oil gave fractions dependent upon the proportion of benzene and paraffin in the mixture. Petroleum rich in benzene and olefine hydrocarbons gave fractions in which the proportions of benzene and olefines increased with the density of the fractions. The explanation is put forward that the fuller's earth acts as a dialysing septum on the petroleum, which is an emulsoid; the paraffin oils have free passage, while the bituminous material is adsorbed and coagulated by the extensive surface of the fuller's earth, and carries with it the sulphur and nitrogen compounds, and the benzene and olefine hydrocarbons. O. E. M.

Estimation of Rosin in Blown Oils. W. Fahrion. (*Chem. Rev. Fett Ind.*, 1913, **20**, 150-152, 177-179.)—Twitchell's method of estimating rosin (ANALYST, 1891, **16**, 169) gives incorrect results when applied to blown oils, owing to the hydroxy acids being incompletely esterified. The error thus produced is reduced, but not eliminated, by making the estimation by the author's modified method (ANALYST, 1911, **36**, 557). In examining blown oils, 5 grms. of the sample (from which any metals have been previously removed) are dissolved in 50 c.c. of petroleum spirit, and the solution mixed with 20 c.c. of 96 per cent. alcohol, and neutralised with $\frac{N}{1}$ alkali solution, with phenolphthaleïn as indicator. It is then brought to an alcoholic strength of about 60 per cent., shaken, and left overnight. The soap solution is diluted to at least 200 c.c., acidified with hydrochloric acid, and twice shaken out with petroleum spirit. The extracts are united and esterified with absolute alcohol and hydrochloric acid, and the rosin estimated as described (Analyst, 1911, **36**, 557), the last traces of soap being removed by washing the solution of the esters with 10 c.c. of 60 per cent. alcohol. These washings are added to the solution of the rosin soap. Esterification of the fatty acids insoluble in petroleum spirit is more difficult in the case of oxidised than of unoxidised oils. Thus, a blown linseed oil containing no rosin yielded by this method 0·86 per cent. of a substance which was apparently rosin. On the other hand, certain neutral bodies and hydroxy-abietic acids which are present in colophony escape estimation by this method. The amount of these substances ranges from about 10 to 25 per cent., with an average of about 14 to 15 per cent. The results of the estimation are also influenced to some extent by the relative proportions of the fatty and rosin acids. The following corrections were found applicable in the case of the samples examined by the author: Amounts below 1 per cent. were ignored, whilst those between 1 and 4 per cent. were regarded as rosin, without making any addition. When above 4 per cent., the results were multiplied by 1·17, an empirical factor corresponding to the average loss of hydroxy-abietic acids and neutral substances in the colophony. C. A. M.

New Colour Reactions of Di- and Tri-Phenols. O. Schewket. (*Biochem. Zeitsch.*, 1913, **54**, 282-285.)—A solution of catechol (0·01 grm.) in 1 c.c. water is treated with 3 to 5 drops of a 1 per cent. solution of iodine in potassium iodide, diluted somewhat with water, and a few drops of 5 per cent. sodium hydroxide solution added, when a beautiful green coloration immediately forms. On standing or boiling, the colour changes to a dirty brown, but after cooling or vigorous shaking, or on adding a few drops of hydrogen peroxide, the green colour reappears. This reaction distinguishes catechol from resorcin and hydroquinone. Pyrogallol similarly treated yields an evanescent bluish-violet to reddish-violet coloration. A solution of pyrogallol in 30 to 50 per cent. alcohol after adding a few drops of sodium or potassium hydroxide gradually yields a violet coloration, very similar to permanganate. This test distinguishes pyrogallol from phloroglucin. On adding a solution of iodine in potassium iodide drop by drop to a solution of phloroglucin (0·01 grm.) in 5 c.c. hot water the colour of the iodine disappears. On adding a few drops of caustic soda to this solution a light brown colour appears, which on boiling turns into a light reddish-violet (distinction from pyrogallol). On adding a few drops of caustic soda to a solution of phloroglucin (0·01 grm.) in 5 c.c. boiling water and shaking, a bluish-violet coloration appears. This reaction, which depends upon oxidation by atmospheric oxygen, succeeds better on adding a few drops of hydrogen peroxide. The coloration produced lasts for days; on acidifying, the colour changes, first into light red and then into light yellow. On making alkaline, the bluish-violet colour reappears. This reaction is sensitive and characteristic of phloroglucin, and distinguishes it from pyrogallol. E. W.

Directions for Testing Pyridine Bases. (*Chem. Zeit.*, 1913, 37, 1035.)—The following directions have been formulated by the German Customs' Board for the examination of pyridine bases :

Colour.—The colour should not be darker than that of a freshly prepared solution containing 2 c.c. of $\frac{N}{10}$ iodine in 1 litre of water. The colours are to be compared in tubes 150 mm. long and 15 mm. wide, closed at the ends with glass plates and screw-caps, having a hole 12 mm. in diameter.



Reaction with Cadmium Chloride.—Ten c.c. of the pyridine bases are dissolved in 1 litre of water, and 10 c.c. of this solution are shaken with 5 c.c. of a solution of 5 grms. of anhydrous fused cadmium chloride in 100 c.c. of water. A copious separation of crystals should appear in ten minutes. The precipitate, collected on a filter of 9 cm. diameter, weighing 0·45 to 0·55 grm., drained, and dried on filter-paper for one hour at 50° to 70° C. without washing, should weigh not less than 25 mgrms.

Reaction with Nessler's Reagent.—Ten c.c. of the same pyridine solution with 5 c.c. of Nessler's reagent should give a white precipitate.

Boiling-Point.—One hundred c.c. of the pyridine bases are measured into a short-necked flask of 180 to 200 c.c. capacity; the flask is placed on an asbestos plate with a circular hole, and a distillation-head, with a bulb of the dimensions shown in the figure, is fitted and connected with a Liebig's condenser having a water-column of at least 40 cm. At the other end an adaptor conducts the distillate into a narrow measuring cylinder of 100 c.c. The bulb of the thermometer must reach to the centre of the bulb in the head of the flask, and the column of mercury must be surrounded by the vapour. Distillation is conducted at the rate of 5 c.c. per minute. When the thermometer indicates 140° C., the flame is extinguished, and the volume of distillate is read when the liquid ceases to drop. The temperature is then raised to 160° C., and the same operation repeated. At least 50 c.c. should distil at temperatures up to 140° C., and 90 c.c. up to 160° C., the distillate being measured at 15° C.

Solubility in Water.—Fifty c.c. of the sample of pyridine bases, when mixed with 100 c.c. of water, should give a clear or faintly opalescent mixture without separation of layers, which, after five minutes and before ten minutes have elapsed since mixing, should be sufficiently transparent in the 150 mm. tube to allow printed characters to be read when the tube is held over them in a vertical position at such a distance that the printing is illuminated.

Moisture.—Twenty c.c. of pyridine bases and 20 c.c. of caustic soda lye, sp. gr. 1·40, are measured by a pipette into a stoppered cylinder, graduated in $\frac{1}{8}$ c.c., and shaken. After settling, the upper layer should measure at least 18·5 c.c. at 15° C.

Titration.—Ten c.c. of pyridine bases are placed in a 100 c.c. flask containing 50 to 75 c.c. of water. The mixture is made up to the mark and shaken. Ten c.c. are then titrated with $\frac{N}{1}$ sulphuric acid, using congo paper as indicator, until a drop shows a distinctly blue edge, which then disappears. Not less than 9·5 c.c. of $\frac{N}{1}$ acid should be required.

J. F. B.

Estimation of Starch in Commercial Products. J. Pieraerts. (*Bull. de l'Assoc. des Chim. de Sucr. et Dist.*, 1913, **30**, 628; through *Chem. Zentralbl.*, 1913, II., 176-177.)—The estimation of starch by converting it into soluble starch and examining the solution in the polarimeter is discussed by the author in the light of his own experiments. Potato starch containing 98·86 per cent. of starch, reckoned on the dry substance, was used, 5 grms. of it being well mixed in a 200 or 250 c.c. measuring-flask with 100 c.c. of the reagent under investigation, and heated in an autoclave for a period not exceeding two hours at 3 atmospheres. After cooling, the liquid was clarified with alumina, made up to the mark, and examined in the polarimeter. The reagents used were: (1) 2·5 per cent. solutions of citric, acetic, succinic, and mono- and trichloro-acetic acids; (2) cold saturated solution of picric acid; (3) 5 per cent. solutions of citric and trichloroacetic acid; (4) salicylic acid, solid, in quantities of 25 per cent. of the solution; (5) asaprol citrate (7·5 grms. asaprol dissolved in 600 to 700 c.c. 25 per cent. citric acid, cleared with a small quantity of animal charcoal, made up to 1 litre with 25 per cent. citric acid, and filtered). The samples of starch were dried in a vacuum at a temperature gradually rising to 120° C. Methods of rendering the starch soluble by means of alkalis, mineral acids, or high pressure, were not considered. The experiments showed that the hydrolysis proceeds differently, as shown by the iodine test, according to the acid employed. Trichloroacetic acid in particular is different from the rest, causing the formation, from the beginning, of *l*-dextrosan, while the other acids give amylodextrin, and to some extent erythrodextrin. The action of acetic, salicylic, and picric acids is not satisfactory, but very accurate starch determinations can be carried out with 2·5 per cent. solutions of citric acid, monochloroacetic acid, or succinic acid, or with asaprol citrate and 5 per cent. citric acid. Substances poor in starch are best treated with 2·5 per cent. citric, succinic, or monochloroacetic acid in an autoclave with the valve open for one hour. The results with tricholoro-acetic acid were all much too low. The asaprol did not equal citric acid in hydrolysing effect; it indeed appeared to delay the further hydrolysis of the amylodextrin. In two hours at 3 atmospheres, 2·5 and 5 per cent. citric acid, 5 per cent. trichloroacetic acid and asaprol citrate effect complete conversion to dextrose, without the formation of considerable quantities of humus-bodies. O. E. M.

Action of Tartaric Acid on Tin in the Presence of Oxygen. A. C. Chapman. (*J. Chem. Soc.*, 1913, **103**, 775-781.)—Rods of pure tin were exposed for long periods of time to the action of 5 and 10 per cent. solutions of tartaric acid by suspending them in bottles nearly full of the acid, and closed with corks through which passed the tin rods in such a manner as to allow of the very slow access of atmospheric oxygen to the liquid. If the fitting of the corks was such as to prevent this, no action occurred, but in the other cases crystals of stannous tartrate were deposited. The stannous tartrate so formed undergoes slow dissociation, and the resulting stannous hydroxide slowly oxidises, forming a coloured compound which is probably hydrated sesquioxide of tin. Orange-coloured colloidal solutions containing as much as 6 per cent. of tin were obtained. H. F. E. H.

INORGANIC ANALYSIS.

Colour Reactions of Alkaline Earths with Oxygallol Derivatives. O. Schewket. (*Biochem. Zeitsch.*, 1913, **54**, 285-291.) — Numerous colour and precipitation reactions are given for solutions of calcium, barium, and strontium salts with alkaline solutions of tannic, gallic, and pyrogallic acids in the presence and absence of alcohol. These reactions are recommended for the detection of the alkaline earths in presence of each other. For the detection of calcium in the presence of strontium and barium, 10 c.c. of a 2 per cent. solution of the earths are treated with a small quantity of pyrogallol and a few drops of caustic soda added, when an intense violet coloration indicates calcium. The alkaline earth solution (10 c.c.) is treated with 5 c.c. alcohol and 10 drops of 1 per cent. gallic acid, and a few drops caustic alkali added; a rose-red precipitate and slow formation of blue colour indicate calcium. In the absence of calcium, strontium can be detected in presence of barium by extracting the mixed sulphates with water and treating the filtrate with tannic acid and caustic alkali. The formation of an evanescent meadow-green colour indicates strontium. E. W.

Quantitative Separation of Chromium and Aluminium: Analysis of Chromite. F. Bourion and A. Deshayes. (*Compt. rend.*, 1913, **157**, 287-289.) —A method was recently described (ANALYST, 1913, 387) for the quantitative separation of the oxides of chromium and iron by conversion into chlorides by the action of a mixture of chlorine and sulphur chloride, the chromium chloride being insoluble. The same process has now been applied for the separation of chromium and aluminium oxides, the reaction in this case being complicated by the circumstances that the aluminium oxide is not so easily chlorinated as ferric oxide, and that aluminium chloride forms a double compound with sulphur chloride. When the aluminium oxide amounts to 50 per cent. or more of the mixture, the loss on heating is very considerable; nevertheless, satisfactory results may be obtained by adding to the oxides a certain amount (*e.g.*, 30 per cent.) of ammonium sulphate. With mixtures containing less than 50 per cent. of chromium oxide, there is always a slight residue unattacked; the whole of the chromium chloride is volatilised. Mixtures containing more than 60 per cent. of chromium oxide behave like the mixtures of chromium and ferric oxides. The double chloride of aluminium and sulphur dissolves in water with evolution of much heat, but without appreciable formation of sulphur. The method has been applied to the analysis of the mineral chromite, which is mainly ferrous chromite, accompanied by oxides of aluminium and magnesium, and a little calcium and silica. For the satisfactory chlorination of the mineral, it must first be broken down by heating, preferably with mercuric sulphate, the temperature being gradually raised to redness. The material so prepared is chlorinated in the manner described, the conversion being complete in about five hours; the silica is not completely attacked. The constituents are thus separated into three groups; the non-volatile residue contains unattacked silica, magnesium and calcium chlorides; the volatilised chromium chloride is insoluble in water, and is collected in a Gooch crucible; the volatilised ferric and aluminium chlorides are readily soluble in water. Each of the constituents is then estimated by ordinary methods, but a quantitative estimation of the silica in this manner is not possible.

J. F. B.

New Separation of Chromium and Manganese. W. Cornelius. (*Pharm. Zeit.*, 1913, **58**, 427; through *Chem. Zentralbl.*, 1913, II., 175.)—The fusion is carried out in the usual manner, and the fused mass, containing potassium permanganate and dichromate, is dissolved in a large quantity of water, and heated on the water-bath with sodium nitrite solution until the separation of hydrated manganese peroxide is complete. The chromium remains in solution, being partly or wholly converted into chromate. The precipitate is washed on a filter with hot water, ignited strongly, and weighed as manganous manganic oxide. The chromate in the filtrate is converted into chromium chloride, with sodium nitrite and hydrochloric acid, precipitated with ammonia, and weighed as chromic oxide. Specimen analyses show the utility of the method.

O. E. M.

Separation of Chromium and Manganese. W. Dederichs. (*Pharm. Zeit.*, 1913, **58**, 446; through *Chem. Zentralbl.*, 1913, II., 175.)—The following method is said to be superior to the method of Cornelius (see preceding abstract) in simplicity and rapidity. The melt of manganate and chromate is converted, by evaporating it to dryness with concentrated sulphuric acid, into the mixed sulphates, which are then converted into sodium manganate and chromate by fusion with sodium hydroxide and a small quantity of sodium peroxide. The fused mass is dissolved in hot water, and sodium peroxide added until the solution is yellow; the hydrated manganese dioxide is then filtered off. The chromium is estimated in the filtrate, after acidifying it with hydrochloric acid, by titration with potassium iodide starch and thiosulphate, or, after neutralising it with nitric acid, by precipitating with mercurous nitrate, igniting, and weighing as chromic oxide.

O. E. M.

Copper in Distilled Water. E. Abel. (*Zeitsch. f. Elektrochem.*, 1913, **19**, 477-480; through *Chem. Zentralbl.*, 1913, II., 479-480.)—The reaction between hydrogen peroxide and sodium thiosulphate is bimolecular. If, however, the water has been distilled from a copper vessel, the catalytic action of the copper in the water upon the reaction is so pronounced that it is possible to estimate the proportion of copper by the velocity of the reaction. Other metals, such as iron, chromium, and manganese, have no appreciable influence upon the reaction.

C. A. M.

Analysis of Ferrotitanium. A. R. Scott. (*Chem. News*, 1913, **108**, 52-53.)—The following scheme of analysis is recommended both on the ground of simplicity, and because it effects a very sharp separation of titanium and aluminium. The finely divided alloy (0·25 grm.) is fused with 10 grms. potassium bisulphate until gritty particles can no longer be felt. The cooled melt is extracted with water in a porcelain basin, 10 c.c. of sulphuric acid being added, and the whole kept hot for half an hour. The insoluble matter is then filtered off, washed, ignited in a platinum crucible, and fused once more with a little bisulphate. The melt is extracted as before, and the silica (contaminated always by a little ferric oxide and titanium dioxide) is filtered off, washed, ignited, and weighed. The residue is treated with hydrofluoric acid and reweighed to determine the silica, and the small residue of non-volatile matter is dissolved in hydrochloric acid and the iron precipitated by means of ammonia. The difference between the weight of non-volatile matter remaining after treatment with hydrofluoric acid and the weight of ferric oxide found gives the weight of titanium dioxide precipitated with the silica.

The combined filtrates from the bisulphate fusions are treated with ammonia until a slight permanent precipitate results. This is redissolved by the least possible addition of hydrochloric acid, and then a solution containing about 15 grms. sodium thiosulphate is added. The mixture is boiled for fifteen minutes, and then filtered through a pulp filter. The precipitate is washed with water acidified with acetic acid, and is finally ignited and weighed as titanium dioxide. The filtrate is concentrated considerably, diluted with 200 c.c. hot water, and this alternate concentration and dilution repeated until a clear solution results. Bromine water is added, and any titanium dioxide which may have passed through the pulp filter is filtered off, washed, ignited, and weighed. The filtrate is heated to boiling, iron and aluminium are precipitated by means of ammonia, and the mixed hydroxides are filtered off, washed, ignited, and weighed as oxides. The ferric oxide is then determined by solution of the mixed oxides in hydrochloric acid, reduction by means of stannous chloride and titration with dichromate, alumina being calculated by difference. In the filtrate from the hydroxides of iron and aluminium, manganese is precipitated by means of bromine, and weighed as Mn_3O_4.

G. C. J.

Estimation of Fluorine in Zinc Ores. L. Schneider. (*Oesterr. Zeits. Berg.- u. Hüttenwesen*, 1913, **61**, 365-367; through *Chem. Zentralbl.*, 1913, II., 615.) —Estimation of fluorine in zinc ores by distillation with concentrated sulphuric acid in the presence of quartz, and collection of the silicon hydroxide which separates in water, gives inaccurate results. The bulk of the fluorine is liberated as silicohydrofluoric acid, but a portion escapes with the sulphur dioxide as hydrofluoric acid. It is therefore necessary to precipitate the fluorine as calcium fluoride after removal of the sulphur dioxide. Ten grms. of the finely powdered ore are mixed with 5 grms. of quartz-powder, and heated on an oil-bath at 160° to 170° C. with about 100 c.c. of strong sulphuric acid, whilst a current of dry air is meanwhile drawn through the apparatus. The gases are passed through a U-tube filled with fragments of quartz into a receiver containing 100 c.c. of water. After the distillation, the water in the receiver is mixed with potassium nitrate and evaporated to dryness, the residue taken up with water, and 25 c.c. of ammonia solution added. The precipitate is separated and ignited in a platinum basin, the residue ($CaF_2 + SiO_2 + CaO$) treated with acetic acid, evaporated to dryness, and ignited, and the calcium fluoride and silica weighed. The silica is then expelled by means of hydrofluoric acid. The weights of calcium fluoride and silica should stand in the empirical ratio of 1 : 3·4. C. A. M.

Detection of Gold and Platinum. G. Malatesta and E. D. Nola. (*Boll. Chim. Farm.*, 1913, **52**, 461-463; through *Chem. Zentralbl.*, 1913, II., 716-717.)—The method devised by Pertusi and Gastaldi for the detection of hydrocyanic acid (by means of copper acetate and benzidine acetate) may be adapted to the detection of gold and platinum. The reagent consists of a solution of 1 grm. of benzidine in 10 c.c. of acetic acid and 50 c.c. of water. With solutions of gold salts it gives a deep blue coloration, which gradually changes to violet. In the presence of free acetic acid the coloration is green, but is changed to blue on the addition of an excess of benzidine. Very dilute platinum solutions yield with the reagent a blue flocculent precipitate, which may possibly be utilised for the estimation of the metal. Heat promotes the separation of the precipitate and makes it darker. Dilute solutions of ferric salts give with benzidine acetate a blue coloration, which is only stable in the presence of excess of benzidine. Free mineral acids do not affect the gold reaction, but retard the precipitation of platinum in the cold. Before applying the test for gold or platinum, iron salts should be removed by means of dilute hydrochloric or nitric acid. The reaction is capable of detecting 0·0000035 grm. of gold and 0·0000125 grm. of platinum. C. A. M.

Colour Reaction of Hypochlorites with Methylaniline and Ethylaniline. P. N. Leech. (*J. Amer. Chem. Soc.*, 1913, **35**, 1042.)—Hofmann (*Ber.*, 1874, **7**, 526; and *Ann.*, 1851, **77**, 130) stated that no colour reaction was given upon the addition of bleaching-powder solution to methyl- or ethylaniline. Pure methylaniline specially prepared by two separate methods was diluted to make a $\frac{1}{100}$ mol. aqueous solution. The intensity of the blue colour which is produced and its time of appearance vary with the concentration. For 1 c.c. of the solution made alkaline with 3 drops to 0·5 c.c. of 6 N sodium hydroxide solution, and treated with 0·5 c.c. to 2 c.c. of half-saturated calcium oxychloride solution, the colour begins to appear in about twelve seconds, and reaches a maximum intensity of navy blue in about thirty-five seconds. After about one minute the colour begins to fade slowly, leaving a yellow solution and a white precipitate of calcium carbonate. As little as 1 mgrm. in 8 c.c. can thus be detected. An excess of alkali, and especially of bleaching-powder solution, is to be avoided. An analogous colour reaction occurs with pure ethylaniline, except that the blue colour goes through green and dark brownish hues before finally leaving a yellow solution. The time required to produce the colour and its persistence are two to three times longer than with methylaniline.

H. F. E. H.

Solubility of Lead Sulphide and Lead Chromate. G. v. Hevesy and F. Paneth. (*Zeitsch. anorg. Chem.*, 1913, **82**, 323-328.)—The authors have made use of the fact that radium D is inseparable from lead to determine the solubility of lead chromate and lead sulphide. A solution was made containing 10^{-6} grm. of radium D and 10 mgrms. of lead chloride. This solution was treated with excess of potassium chromate, and the resulting precipitate filtered off and washed. A solution of the chromate, saturated at 25° C., was then prepared, filtered from undissolved chromate, and a measured portion was evaporated to dryness, and the radio-activity (β) of the residue measured as soon as equilibrium between radium D and E was established. From this result and the known radio-activity and percentage of lead in the solution which was used to prepare the chromate the solubility of lead chromate at 25° C. was calculated to $1{\cdot}2 \times 10^{-5}$ grm. per litre.

In a similar manner lead sulphide was shown to be soluble to the extent of 3×10^{-4} grm. per litre at 25° C. in pure water, the solubility in water saturated with hydrogen sulphide being just half as much. G. C. J.

Colorimetric Estimation of Small Quantities of Manganese in Water. E. Schowalter. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1913, **26**, 104-108.)—An investigation of the method proposed by Haas (ANALYST, 1913, 225) showed that chlorides are not eliminated from the water by heating with sulphuric acid and persulphate. As it is essential that all hydrochloric acid be removed before the manganese is estimated, the author adopts the method given below. The removal of chlorides by the addition of silver nitrate is objectionable owing to the turbidity produced. At the same time, the presence of a small quantity of silver nitrate renders possible the estimation of manganese in waters containing less than 2 mgrms. of the metal per litre. The reaction is dependent on the concentration of the acid and the quantity of silver nitrate, and in the presence of the latter the full intensity of the permanganate coloration is attained. One hundred c.c. of the water are acidified with 10 c.c. of nitric acid and evaporated to dryness; the residue is treated with 10 c.c. of nitric acid, again evaporated to dryness, and then dissolved in 5 c.c. of nitric acid and 10 c.c. of water. The solution is filtered, the insoluble portion is washed, and the filtrate (measuring not more than 35 c.c.) is heated to boiling after the addition of 5 c.c. of nitric acid and 1 grm. of ammonium persulphate. Ten drops of a 5 per cent. silver nitrate solution are now added and the mixture again boiled, the boiling being maintained for 1·5 minutes after the first appearance of the pink coloration. The intensity of the coloration obtained is then compared with that of standard permanganate solutions. The presence of iron salts in moderate quantity, and of organic reducing substances, does not interfere with the estimation.

W. P. S.

Estimation of Mercury in the Air of Rooms, etc., where the Metal is Used. A. Blomquist. (*J. Pharm. Chim.*, 1913, **7**, 8-15, 71-75, 112-118, 166-172.)—The author has estimated the quantity of mercury present in the atmosphere of rooms in laboratories, hospitals, and works, where considerable quantities of the metal or its salts are employed. It was found that mercury vapour was almost invariably present in such places, the quantity varying from a mere trace to about 1 mgrm. per 4,000 litres of air. Mercury was also detected in the dust collected from the rooms, and was present in the urine of persons working therein. The method employed for the estimation of the mercury consisted in liberating chlorine in the room, and then aspirating 4,000 litres of the air through a series of absorption vessels containing a dilute hydrochloric acid solution of stannous chloride and potassium hydroxide solution respectively. The contents of the vessels were then mixed and treated with hydrochloric acid and potassium chlorate. The mercury in the solution was then precipitated (amalgamated) by the addition of zinc dust, redissolved, and collected on a gilded asbestos filter as described by Farup (ANALYST, 1913, 111). In the case of minute quantities of mercury, it was found to be preferable to collect the mercury from its solution on a small piece of brass-foil. The latter was then heated in a tube, and the mercury collected in a capillary portion of the latter. The small sublimates thus obtained were compared with capillary tubes containing known quantities of sublimed mercury. W. P. S.

Sensitive Reaction of Molbydenum. A. Komarowsky. (*Chem. Zeit.*, 1913, **37**, 957.)—In Melikoff's test for molybdenum the solution is evaporated to dryness on the water-bath, and the cold residue treated with a drop of strong ammonia and a drop of hydrogen peroxide. In the presence of molybdenum, a cherry-red to rose-yellow coloration, due to the formation of ammonium permolybdate, is obtained. The test is capable of detecting 0·006 mgrm. of molybdenum. Ammonium permolybdate is decomposed by gentle heat, and the original substance reproduced. C. A. M.

Determination of Phosphorus in Steels containing Vanadium. J. R. Cain and F. H. Tucker. (*J. Ind. and Eng. Chem.*, 1913, **5**, 647-650.)—When vanadic acid is present, even in small quantity, the precipitation of phosphorus as phosphomolybdate is incomplete, and the precipitate is contaminated by vanadium compounds. The following method, which depends on reducing vanadium compounds to the quadrivalent condition and preventing their reoxidation until the precipitation of the phosphorus is complete, is adapted to any steel containing vanadium, provided no other element which ordinarily complicates the determination of phosphorus is present. If tungsten, titanium, arsenic, tin, etc., are present, their disturbing influence is eliminated by the usual methods for steel containing no vanadium. Neither nickel, copper, chromium, molybdenum, nor aluminium, interfere.

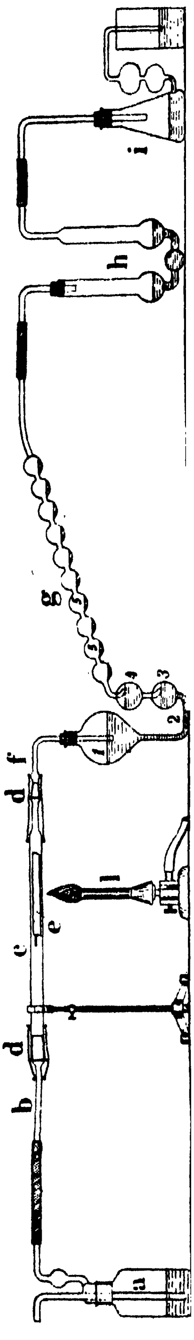
The steel (1 to 2 grms.) is dissolved in 100 c.c. of dilute nitric acid (sp. gr. 1·135), the solution is oxidised while hot by a slight excess of permanganate, manganese dioxide or excess of permanganate is reduced by means of sulphurous acid, and the solution is cooled and partially neutralised by addition of 40 c.c. of dilute ammonia (sp. gr. 0·96). The solution is cooled again to 15° to 20° C., and 5 c.c. of a saturated solution of ferrous sulphate and a few drops of sulphurous acid solution are added to complete the reduction of the vanadium. Molybdic reagent (40 c.c.) is added, and the mixture shaken vigorously for ten minutes, after which the precipitate is filtered off, washed until free from acid, and titrated by the alkalimetric method.

The use of a second reducing agent (ferrous sulphate) can be avoided, and the complete reduction of the vanadium effected by means of sulphurous acid; but it is essential to keep the temperature low, and reduction is then so slow that the method is not recommended. On the other hand, although ferrous sulphate alone would reduce excess of permanganate as well as the vanadium, it is found that, unless sulphurous acid is simultaneously present, the reduced solution suffers reoxidation before the precipitation of the phosphorus can be effected. It is probable that the nitric oxide formed by the action of the ferrous salt on the nitric acid acts as a carrier of atmospheric oxygen to the vanadyl compound. Whatever the nature of the action, the test numbers show that it is completely held in check by the presence of a trace of sulphite. G. C. J.

Action of Persulphate on Iodate, and Estimation of Periodate in Presence of Persulphate. E. Müller and W. Jacob. (*Zeitsch. anorg. Chem.*, 1913, **82**, 308-314.)—In absence of iodate, a mixture of periodate and persulphate may be analysed as follows: To a measured volume of the solution is added so much caustic alkali that the mixture is approximately $\frac{N}{1}$ alkaline. Potassium iodide is added, the mixture boiled for two minutes, cooled, acidified with dilute sulphuric acid, and the liberated iodine is titrated with $\frac{N}{10}$ thiosulphate. This measures the sum of the available oxygen of the persulphate and periodate. Another portion is made alkaline with caustic alkali, and reduced by boiling for an hour with zinc under a reflux air-condenser. The iodide in the resulting solution of iodide and sulphate is then determined by pouring off from the zinc, cooling, acidifying with dilute sulphuric acid, treating with excess of iodate, and titrating with $\frac{N}{10}$ arsenious solution after addition of a considerable excess of sodium bicarbonate. Five-sixths of the iodine found is the measure of the periodate originally present, and the oxygen equivalent of this periodate subtracted from the total available oxygen due to periodate and persulphate gives the measure of the persulphate.

The authors' work was undertaken for the purpose of studying the reaction between persulphate and iodate, one of the products of which is periodate. They have been unable to devise an exact method for the estimation of periodate in presence of persulphate and iodate, but the following method gives results which are ±5 per cent. The solution is made $\frac{N}{2}$ to $\frac{N}{1}$ with caustic alkali, an excess of potassium iodide is added, and the mixture left for twenty-four hours at room temperature. Under these conditions persulphate alone is reduced quantitatively to sulphate with production of iodate, whereas periodate alone is unacted on by iodide in caustic alkaline solution. The error of the method is due to the fact that in presence of persulphate an uncertain proportion of the periodate reacts with iodide to form iodate. After standing twenty-four hours as described, the solution is mixed with a measured and more than sufficient quantity of arsenious solution, then with a large excess of sodium bicarbonate, heated for five minutes to 50° C., cooled, and the excess of arsenious solution titrated with $\frac{N}{10}$ iodine. G. C. J.

Separation of Tin from Tungsten Electrolytically. W. D. Treadwell. (*Zeitsch. Electrochem.*, 1913, **19**, 381-384; through *Chem. Zentralbl.*, 1913, II., 85.)—The chief difficulty in working out a satisfactory method for the electrolytic separation of tin and tungsten has been to find an alkaline electrolyte, from which the tin could be deposited rapidly and in adherent form whilst the tungsten remained in solution as tungstate. A warm solution of sodium sulphide has been found the most satisfactory electrolyte, and the best results are obtained by working at a temperature of 50° to 60° C., and a P.D. of 1·7 to 2·3 volts. The method is not available n presence of molybdenum. G. C. J.



Estimation of Sulphur in Pyrites and Other Sulphides. M. Dittrich. (*Zeitsch. anorg. Chem.*, 1913, **83**, 27-32.)—The method described consists in combustion of the sulphide in a current of oxygen, absorption of the sulphur trioxide and oxidation of sulphur dioxide by means of bromine-water and bromine, and precipitation of the sulphuric acid by means of barium chloride in the usual manner. The combustion is carried out in a specially-constructed silica combustion-tube, 30 cm. long and 12 mm. in internal diameter. The back end is ground to receive the enlarged end of a silica tube of small bore which connects to a sulphuric acid wash-bottle, and thence to the supply of oxygen. The forward end of the combustion-tube is drawn out and ground to fit into the enlarged end of a silica bend, which, except at the enlargement, is 5 mm. in internal diameter. The absorption apparatus *g* is of such design that, if 2 c.c. of bromine be introduced into the large bulb together with the bromine-water, every bubble of gas will come in contact with bromine in the horizontal tube 2, whereas very little bromine vapour will go forward. The Peligot tube *h* is also charged with bromine-water, the Volhard bubbler *i* with caustic soda solution, and *k* with water. The substance to be burned is weighed out into a porcelain boat, which is pushed up to the front of the combustion-tube, and the current of oxygen is so adjusted that two or three bubbles pass per second. The forward part of the boat is then heated with the full flame of a Bunsen burner until combustion commences, when the heat is moderated and the rest of the boat and of the combustion-tube are heated up to prevent condensation of sulphur trioxide. When combustion slackens, the heat is increased, and finally a Teclu burner is used. Finally the flame is carried to the forward ground joint, and beyond it to the bend of the tube *f*. The contents of *g* and *h* are now transferred to a beaker, together with the washings of *f*, boiled to expel bromine, neutralised with ammonia, acidified with 1 c.c. of hydrochloric acid, and heated to boiling. Boiling solution of barium chloride is then added, and the analysis completed in the usual manner. The soda solution in *i* and the residue in the boat should be tested for the presence of sulphate. In the case of pyrites, none will be found, according to the author's experience. With blende, on the other hand, 1 to 2 per cent. of the sulphur will remain in the boat unless the sample be very finely divided and spread in a very thin layer in the boat. A 5-inch boat is necessary to give sufficient distribution to 0·7 grm. of blende.

G. C. J.

Application of Tantalum Electrodes to Electro-Analytical Estimation of Copper and Zinc. G. Wegelin. (*Chem. Zeit.*, 1913, **37**, 989.)—Brunck (*Chem. Zeit.*, 1912, **36**, 1233) and others have recommended the use of tantalum electrodes for the estimation of copper, of zinc from alkaline zincate solution, and for other purposes. The resistance of tantalum towards chemical reagents, including concentrated nitric acid and mixtures of dichromate with sulphuric acid, is high, and its low cost compared with platinum would justify its use, even at some loss of speed or increased complexity of technique. So far as the estimation of zinc is concerned, however, the author has been unable to obtain adherent deposits. No modification of the character of the electrolyte or other conditions of experiment gave rise to results even approximately quantitative. The existence of a superficial layer of oxide, such as forms on aluminium, is held to be the probable explanation of these results. Copper can be deposited quantitatively from sulphuric acid solution, using the current for a single accumulator. Deposition is complete in nineteen hours, but not in fifteen hours, when 0·2 grm. of copper is present in 100 c.c. The relative slowness of the deposition is the only disadvantage of the method, and in some circumstances this disadvantage may be negligible. Attempts to increase the speed of deposition by resort to higher temperatures led to the formation of deposits which were insufficiently adherent. Before use, the electrodes are treated with a mixture of dichromate and sulphuric acid, any copper from a previous estimation being first dissolved in nitric acid.

G. C. J.

Estimation of Thiosulphate in Presence of Sulphites. A. A. Besson. (*Chem. Zeit.*, 1913, **37**, 926.)—In a mixture containing sodium sulphite, bisulphite, and thiosulphate, the sulphite and bisulphite are estimated by the method of Bosshard and Grob (ANALYST, 1913, 297), the sulphite by titration with standard acid and methyl orange, and the total bisulphite by titration of the same solution with standard alkali and phenolphthaleïn, the true amount of bisulphite being calculated from these results. To determine the thiosulphate, another portion of the solution is heated with 25 c.c. of $\frac{N}{10}$ alkali and 20 c.c. hydrogen-peroxide solution (5 c.c. of Merck's perhydrol diluted to 100 c.c.) for ten minutes on the water-bath or wire gauze, and, after cooling, the excess of alkali is determined with methyl orange and acid. The quantity of alkali absorbed by the bisulphite is known and is deducted. One c.c. of $\frac{N}{10}$ alkali corresponds to 0·01241 grm. $Na_2S_2O_3, 5H_2O$.

O. E. M.

Separation of Tungsten from Thorium, Lanthanum, Cerium, Erbium, Didymium, and Silica. M. Wunder and A. Schapira. (*Ann. Chim. anal.*, 1913, **18**, 257.)—The methods were tried on the oxides mixed in known proportions. The quantities recovered showed satisfactory agreement with those taken.

Separation of Tungsten and Thorium Oxides.—The oxides are heated to constant weight and fused for an hour with fifteen times their weight of sodium carbonate; the melt is boiled for twenty minutes with water and more sodium carbonate, and filtered. The residue, thorium dioxide, is ignited; the tungstic trioxide is precipitated from the neutralised filtrate with mercurous nitrate solution containing a slight excess of nitric acid. The thorium dioxide residue is insoluble in 5 per cent. hot hydrochloric acid.

Separation of Tungsten and Lanthanum Oxides.—The method is identical with the above. The residue of lanthanum oxide is completely soluble in hot 5 per cent. hydrochloric acid.

Separation of Tungsten and Cerium Oxides.—The mixture of oxides is volatile, consequently, in testing the validity of the method it was necessary to ignite them separately to constant weight before mixing them. The separation is identical with the above.

Separation of Tungsten and Erbium Oxides.—The filtration of the dissolved melt must take place in the cold. The residue of erbium oxide is dissolved in 5 per cent. hydrochloric acid and reprecipitated with ammonia.

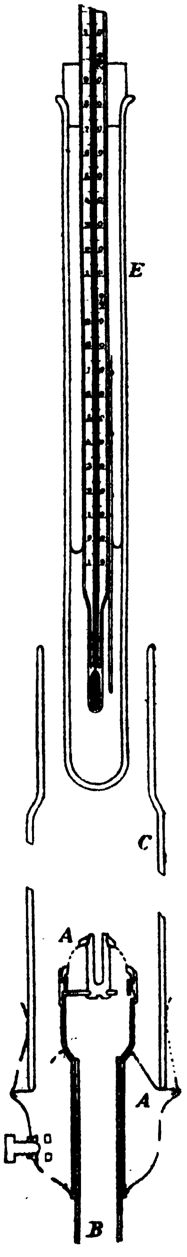
Separation of Tungsten and Didymium Oxides.—In testing the method, the oxides required treating separately to constant weight. The separation is identical with that for tungsten and erbium.

Separation of Tungstic Trioxide, Alumina, and Silica.—In presence of a large excess of ammonium nitrate the tungsten is not precipitated by mercurous nitrate, and the silica, in presence of tungstic trioxide, is only incompletely precipitated by the ammonium nitrate. The separation is therefore effected as follows: The alumina and part of the silica are precipitated with ammonium nitrate, and the precipitate ignited and weighed, treated with hydrofluoric acid and a few drops of sulphuric acid, ignited, and weighed as alumina, the difference giving the silica precipitated with it. The tungsten is precipitated as mercurous tungstate from the filtrate with mercurous nitrate, and treated with hydrofluoric acid and sulphuric acid, the tungstic trioxide and silica precipitated with it being thus obtained. Finally, the rest of the silica is determined by the usual methods in the filtrate from the mercurous tungstate precipitate. O. E. M.

APPARATUS, ETC.

Ebullioscope of High Accuracy. A. Contassot. (*Ann. Chim. anal.*, 1913, **18**, 273.)—The apparatus is designed for the determination of the alcohol-content of wines and other liquids. Previously it has been necessary to determine separately the boiling-point of the water and of the sample, the two operations requiring twenty to twenty-five minutes; an estimation can now be carried out in five to six minutes. The sample is taken as it stands, or diluted, according to its alcohol content. The alcohol in distillery residues and in vinegar in process of manufacture may be determined by means of the apparatus. It is obtainable from A. Manoncourt, 76, Boulevard St. Germain, Paris. O. E. M.

Apparatus for Gas Analysis Laboratories at Coal Mines. G. A. Burrell and F. M. Seibert. (*U.S. Bureau of Mines, Technical Paper* 14, pp. 24.)—An apparatus for the exact determination of carbon dioxide and methane, another for for the exact determination of methane alone, portable modifications of each, and a modified Orsat apparatus for complete approximate analysis of mine air are illustrated and described. No new principle is involved in any of them, but advantage has been taken of the work of Haldane and others to devise appliances exactly suited to the requirements of some mine laboratories. The pieces of apparatus described appear to be identical with some of those in an earlier paper (ANALYST, 1912, **37**, 288), but whereas the journal in which that paper appeared is not everywhere available, the present publication may be obtained free by applying to the Director, Bureau of Mines, Washington, D.C. G. C. J.

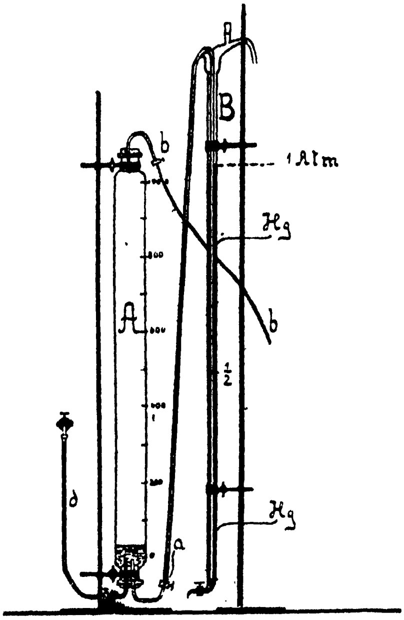


Melting-Point Apparatus for High Temperatures. E. Schwinger. (*Monatsh. für Chem.*, 1913, **34**, 977-979.)—On the upper end of a Bunsen burner, *B*, is fixed an incandescent gas-burner, *A*, as shown in the illustration—if necessary, the tube of the burner being wrapped round with asbestos-paper. On the incandescent burner is fixed the ordinary chimney *C*, of Jena glass provided for that type of burner. This cylinder serves as an air-bath which is very easily regulated, and in which the melting-point apparatus is heated. This consists of the fairly thick-walled test-tube, *E*, 18 to 20 cm. long, held by a clamp at its upper end. The tube forms a bath, charged for high temperatures with an equimolecular mixture of sodium and potassium nitrates, or for lower temperatures with sulphuric acid. With such a form of bath there is plenty of room for expansion, so that it is not necessary to pour away the salt mixture when the operation is finished. With a short thermometer the mercury column may be completely surrounded by the fused salt, and the corrected melting-point directly obtained. The thermometer is fixed in the tube by a split cork. The chief advantage of this apparatus is its convenient and rapid manipulation, so that the temperature of the bath immediately responds to any adjustment of the flame, the heating being performed by the upward current of air. The regulation is facilitated by attaching a pointer to the tap of the burner and a fixed scale. This mode of heating may be applied for other purposes—*e.g.*, maintaining liquids at gentle ebullition for long periods—and temperatures up to 550° C. are readily obtained. J. F. B.

Preparation of Sections of Crystalline Substances. E. Korreng. (*Zentralbl. Min. u. Geol.*, 1913, 408-412; through *Chem. Zentralbl.*, 1913, II., 614.)—Thin sections of fused salts, etc., for crystallographic examination are prepared by rubbing one surface upon emery-paper of different grades and polishing it upon smooth glass, a drop of castor-oil being added in the case of hygroscopic substances. The polished surface is attached to the object-glass with Canada balsam, and the other surface rubbed down with emery-paper until sufficiently transparent, after which the preparation is mounted in Canada balsam. Very hygroscopic substances may be melted between two cover-glasses, which are afterwards mounted in Canada balsam.

C. A. M.

Simple Modification of Skita's Hydrogenising Apparatus. H. H. Franck. (*Chem. Zeit.*, 1913, **37**, 958.)—The measuring cylinder *A* of this apparatus is 75 to 100 cm. long, and has a capacity of about a litre. The upper end is closed by a rubber cork (wired down), through which passes a tube, *b*, which can be connected with the hydrogen cylinder or the shaking vessel; whilst the rubber cork in the lower end has two outlets: one of these, *d*, is attached to the water-tap, and the other, *a*, with a mercury regulator, *B*. In using the apparatus, all air is first expelled from *A* by means of water from *d*. The tube *b* is next connected with the hydrogen cylinder, the tube *d* disconnected from the water-tap, and the gas introduced into *A* until nearly the whole of the water has been driven out. The tube *d* is then again connected with the water-supply, and hydrogen passed into the cylinder until the first bubbles of water rise through the mercury in *B*—*i.e.*, until the pressure equals 1 atmosphere. The tap *a* is now closed, the tube *b* connected with the shaking vessel, containing the unsaturated substance, the tap *a* opened again, and water introduced through *d* until bubbles again appear above the mercury. In this way the absorption is compensated and constant pressure maintained. As the water rises in *A* during the absorption the mercury regulator is raised, or an empirical correction may be applied. The hydrogen value of unsaturated bodies is measured by the volume of gas absorbed.



C. A. M.

New Designs for Specific Heat Apparatus. A. R. Johnson and B. W. Hammer. (*J. Amer. Chem. Soc.*, 1913, **35**, 945-948.)—Two pieces of apparatus are described and illustrated, having for their object the rapid yet accurate determination of the specific heat of liquids. Both depend on electrical heating, and one is intended for use where electrical energy at constant voltage is available, whereas the other is independent of the voltage.

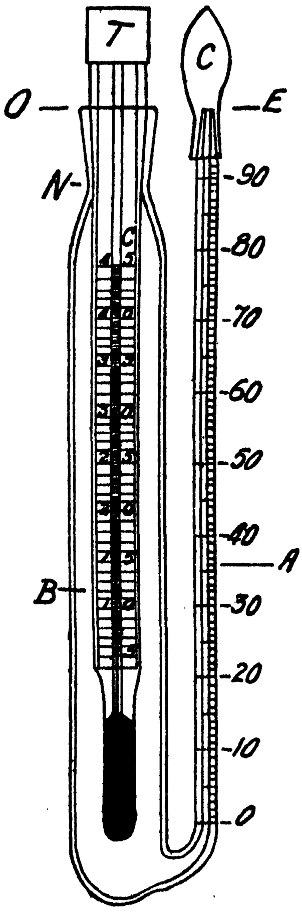
In the apparatus for constant voltage, the calorimeter vessel is a thin copper or glass cylinder of 500 c.c. capacity, which is supported on cork points in a Dewar vessel some 12 cm. in diameter and 15 cm. deep. The Dewar vessel rests on a wooden support and has a hollow wooden cover, which is adapted to be raised and which carries a thermometer and an 8 c.p. lamp which dip into the calorimeter vessel when the cover is in place. The lamp has agitating vanes attached to it, and is adapted to be rotated. The liquid to be tested is placed in the calorimeter, the temperature noted, the current switched on, and readings of the temperature taken every minute for five minutes. A second experiment with an equal weight of water is then made. The correction to be applied for radiation is determined in the usual way. An alternative method for determining specific heats with this apparatus consists in noting the time required to raise a definite weight of the liquid through a definite temperature interval, and comparing this with the time required to raise an equal weight of water through the same interval.

In the second apparatus the calorimeter vessel is a copper or glass cylinder 6·25 cm. in diameter and 8·75 cm. high, and it is supported on cork points in a cylindrical space hollowed in a block of cork about 12 cm. cube. Surmounting this hollowed block is a heavy asbestos cover in three pieces, the middle piece being adapted to slide, and above the asbestos cover is another large block of cork with a cylindrical hole about 5 cm. in diameter bored through it. In the cylindrical space thus formed and resting on the asbestos slide is the heater, consisting of a copper vessel 4·7 cm. in diameter and 8·1 cm. high, containing 100 c.c. of water and, immersed in it, a small incandescent lamp. The heater has a tight-fitting cover, and the leads from the lamp pass through a glass tube which serves as a handle by means of which the whole heating appliance can be raised or lowered. The heater as well as the calorimeter is supplied with a thermometer. In use, 100 grms. of the sample is placed in the calorimeter, and the heater (outside the instrument) is raised to a temperature about 25° C. higher than initial temperature of the sample, and is then placed in its chamber in the upper cork block. When radiation becomes regular, the calorimeter thermometer is read, and when the heater has cooled to some chosen temperature it is dropped into the calorimeter vessel. The liquids in both vessels are agitated by means of vanes attached to the thermometers, and the maximum calorimeter temperature is noted. A similar experiment is then made with water. Corrections for radiation and for the water equivalent of the calorimeter must, of course, be applied.

In a series of determinations of the specific heat of aniline, made with the apparatus first described, no result differed by more than 0·2 per cent. from the mean.

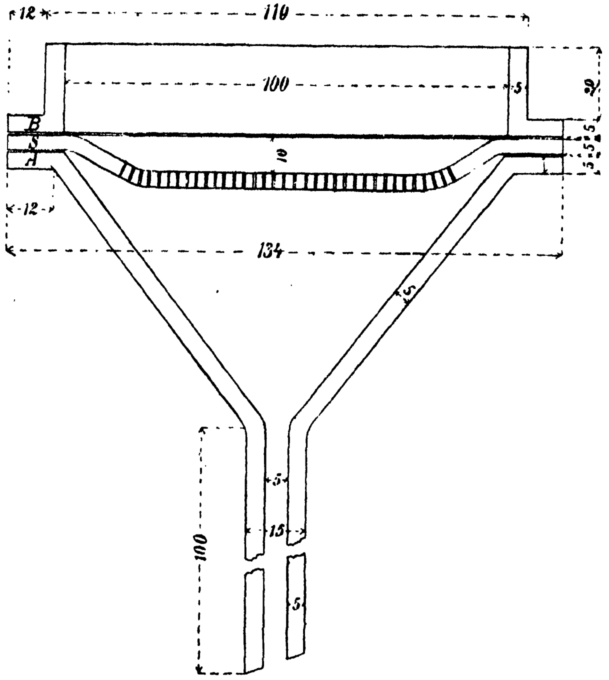
G. C. J.

Combination Specific Gravity Bottle and Dilatometer. C. A. Browne. (*J. Amer. Chem. Soc.*, 1913, **35**, 955-958.)—The accompanying block makes clear the principle of the instrument, which holds about 30 c.c. When used to determine specific gravities it is used like an ordinary pycnometer. By raising or lowering the temperature after the vessel is filled, the coefficients of expansion or contraction may be calculated. The author has found the apparatus convenient for measuring the contraction which sugar solutions undergo during hydrolysis or inversion, and, from the fact that 100 c.c.



of 1 per cent. cane-sugar solution undergo a contraction, during inversion, of 0·02 c.c., the approximate percentage of cane-sugar present in a solution may be measured after the addition of invertase or acid. The contraction which sugar solutions and water undergo when mixed may be easily demonstrated by placing a few c.c. of strong sugar solution in the instrument, filling carefully with water to some mark on the capillary, and then mixing by reversing the whole. H. F. E. H.

Simple Apparatus for Ultrafiltration. R. Zsigmondy. (*Zeitsch. angew. Chem.*, 1913, **26**, 447-448.)—In conjunction with others, the author has previously described (Analyst, 1912, **37**, 223) an apparatus which serves well, but has the disadvantage that the collodion films are liable to get torn at the edges. The apparatus now figured is in three pieces, of which the funnel tube *A* and the ring *B* are of glass, the perforated plate *S* of hard rubber. The whole apparatus was at first constructed of hard rubber, but the partial substitution of glass reduces the cost of construction. The flanges of all three parts are ground to fit, and are held together by clamps when the filter has been prepared. To do this, *B* is removed, a filter-paper is fitted over *S*, including the flange, and on this is superimposed a collodion film. *B* is then set in place, and the whole screwed together. The collodion films are prepared by diluting 4 parts of commercial collodion (6 per cent.) with 2 parts ether and 5 parts alcohol, pouring on to a polished glass plate, and stripping in the manner previously described. By the aid of a pump 100 c.c. of water may be sucked through a 9 cm. filter in five minutes, and 100 c.c. of water separated from 0·1 grm. of colloidal ferric hydroxide in twenty minutes. G. C. J.



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REPORT.

Metropolitan Water Board: Seventh Annual Report, March 31, 1913. A. C. Houston.—The author considers that the means now adopted for the purification of the Metropolitan water-supply are such as to render the water reasonably, if not absolutely, safe for drinking purposes. The average quality of the water supplied during the year, together with that of the Rivers Thames and Lea, was as follows:

Parts per 100,000.	Thames Water.		Lea Water.	
	Raw.	Filtered.	Raw.	Filtered.
Ammoniacal nitrogen	0·0051	0·0004	0·0088	0·0003
Albuminoid „	0·0146	0·0063	0·0135	0·0053
Oxidised „	0·26	0·24	0·30	0·26
Chlorine	1·56	1·53	2·0	1·96
Oxygen absorption, 80° F. ...	0·2097	0·0924	0·190	0·0753
Total hardness	22·3	21·16	25·12	23·37
Permanent hardness	4·96	5·58	5·99	6·36
Turbidity	2·57	nil	3·12	nil
Colour: mm. brown in 2 feet...	65	18	67	16
Bacteria per c.c. gelatine ...	6550	14·5	11,772	30·5
„ „ agar	428	1·9	484	5·2
„ „ *B. coli* ...	15	2 to 3 per l.	4	2 to 3 per l.

Owing to the excessive development of *Tabellaria* in the Staines reservoirs, the filtered water from these reservoirs had often a most unpleasant "geranium" taste and smell. Although this was innocuous, the water was at times almost undrinkable.

The author concludes with a review of the sources of the London water-supply, in which he states that the river waters are undoubtedly unsatisfactory in quality, and the judicious selection of water is becoming increasingly difficult, as the consumption increases whilst the volume of the rivers remains constant. The levelling effect of storage, however, renders the possibility of any sudden dangerous pollution a matter of less grave concern than would otherwise be the case. But if, owing to any accidental circumstance, a considerable amount of infective material should gain access to the water directly feeding the filters, the result would be likely to be disastrous. "In this connection, too much reliance must not be placed on the work of water examination. For technical and other reasons, the most perfect and costly system of water examination that could be devised might fail altogether to detect an accidental contamination." J. H. J.

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REVIEWS.

AN INTRODUCTION TO THE STUDY OF FUEL. By F. J. BRISLEE, D.Sc. London: Constable and Co., Ltd. Pp. xxii + 269. Price 8s. 6d. net.

This is the first of a series of volumes on "Outlines of Industrial Chemistry,' under the editorship of G. D. Bengough, and has for a subtitle "A Textbook for those entering the Engineering and Technical Industries." The series is intended, in the words of the editor, to provide a link between the elementary chemistry taught in schools and colleges which provide instruction up to, say, the matriculation standard of a modern University, and the larger textbooks dealing with the details of the several industries.

The subject of fuel is of importance in a wide range of industries, and there is no doubt that the editor has made a wide choice of subjects for the opening volume, more especially as there was no modern work on the subject covering the same ground. The arrangement of the book is good, the exposition of the subject being clear and lucid, proving the author's experience as a teacher; but as a whole the book is disappointing. One of the most usual complaints of the manufacturer employing a man straight from the University is that he is apt to waste time in working out his results to five places of decimals when his fundamental measurements are not reliable to three. A striking example of this is given in chapter viii., where the heats of formation of two coals are worked out. Analyses of two coals are given, from which the heats of combustion are calculated as 6301·68 and 6183·65 respectively; the measured heats of combustion are taken as 7433·00 and 6013·00 calories; and the differences of these are quoted as the heats of formation. Incidentally it may be noted that the analyses of the coals, although apparently complete, including carbon, hydrogen, oxygen, nitrogen, moisture, sulphur, and ash, add up to 98·65 per cent. and 100·52 per cent., and are probably liable to sampling errors of at least 0·5 per cent. And yet we have a thermal measurement given to *six* significant figures. This example is quoted from Juptner, but there are plenty of other examples elsewhere. Thus, 11,934 B.T.U. per pound is given as a figure obtainable with the W. Thomson calorimeter. In chapter ii., again, on the weight and volume of air required for combustion, starting with the round number of 21 per cent. of oxygen in air by volume and a fuel analysis of three significant figures, we find the weight and volume of the products of combustion given as 12,856·5 grms. and 9410·2 litres. On p. 221 there is another example, in which the weight of the products of combustion of a coke is given as 11,193·4 grms., or, including excess air, 22,511·8 grms.

Returning to the narrower standpoint of analysis, with which this journal is more specially concerned, very little stress is laid on the importance of sampling, nor is there any intimation of the magnitude of the errors likely to arise from this source. The methods given for the determination of moisture and volatile matter in coal would lead to figures differing considerably from those obtained by methods currently used. In flue gas analysis the Winkler burette is described as a Hempel; both the methods given for the estimation of carbon monoxide in flue gas are unreliable, the accurate iodine pentoxide method not being mentioned. The Stead gas sampler is figured, but the instructions for its use are quite inaccurate, the author not having, apparently, grasped the reasons for its design. No carbon dioxide recorder is figured, for lack of space, but eleven pages are devoted to a full mathematical treatment of producer gas and water gas reactions. This chapter concludes with the remark: "From a technical standpoint, however, these calculations are at present of little value, as equilibrium is certainly not reached in the production of either water gas or producer gas." The reviewer agrees with this conclusion, and thinks this chapter might be omitted with advantage.

In chapter iv. descriptions and working directions are given for the Lewis Thompson, W. Thomson, Rosenhain, F. Fischer, Berthelot-Mahler, and Parr coal calorimeters, also for the Junker and Boys gas calorimeters. The descriptions are clear and the instructions sufficiently detailed for use by a student. There is no attempt, however, at a critical examination of the various types, and the student would naturally, but erroneously, assume that all were of equal accuracy. It is difficult to fathom the reason which has led the author to classify the Lewis Thompson instrument as belonging to the type in which the combustion takes place at atmospheric pressure, whilst the Parr (sodium peroxide) calorimeter is classified under the type in which combustion is carried out at pressures greater than atmospheric. There is no discussion of the use of the "gross" and "net" calorific values; the latter is defined incidentally as obtainable "by subtracting the latent heat of vaporisation of water, 10,800 cals. per molecule, from the value obtained experimentally."

The chapter on natural solid fuel contains some curious statements. Anthracitic coals and anthracites are described under the main heading of "Bituminous Coals." The calorific values of the coals given on pp. 132 to 135 are impossible, and in no way correspond with the compositions given. It is difficult to understand how anyone who has had anything to do with the handling of fuel could give 16,560 to 17,280 B.T.U. per pound as a typical value for short-flame caking coals. These figures are repeated in the table on p. 135. The figures in the adjacent column, on the other hand, headed "Water evaporated per Cent." (probably a misprint for "Pound"—"Water evaporated per Pound") are much too low, and do not correspond to the average boiler efficiency. In this chapter also it is stated that "anthracite coal is used for firing the boilers of steamships, especially warships."

The book does not appear likely to be of service to the class of students mentioned in the preface. G. NEVILL HUNTLY.

METHODS IN CHEMICAL ANALYSIS ORIGINATED OR DEVELOPED IN THE KENT CHEMICAL LABORATORY OF YALE UNIVERSITY. Compiled by F. D. GOOCH. New York: John Wiley and Sons; London: Chapman and Hall, Ltd. 1912. Price 17s. net.

As stated in the preface, the object of this volume is to present concisely the principal results reached by workers in the Kent Chemical Laboratory in the investigation and development of methods in (inorganic) chemical analysis. Consequently one finds here described apparatus and processes with many of which one has become familiar through the original papers or abstracts published in the ANALYST during the past twenty years or more. As representing the work of a laboratory presided over by a master in the science of analytical chemistry, it is valuable to have these results collected in a single volume, and every practising analyst will find it worth while to place the volume upon his shelves. It is full of original ideas in apparatus and methods, the descriptions are models of clearness, and the illustrations are beautiful examples of the book-illustrator's art. It is unfortunate that, in order to obtain a surface suitable for their reproduction, it should have been necessary to load the paper with mineral matter to such an extent as to make the volume inconveniently heavy, but this is a fault by no means limited to the volume under notice.

References are given to the originals of all the papers, there is a good index of authors, and a fair one of subject-matter. In the first line of the table on p. 242 there appears to be one of the very few errors noticed by the reviewer—the CaF_2 present was presumably nil. On p. 203 "dessicator" is spelt with two *s's* and one *c*; on p. 227 one *s* and two *c's* are correctly used. "Etherial" is printed for "ethereal" on p. 492, and throughout the book "cm^3" is used for "c.c.," which, although sanctioned, seems cumbersome. These are details. The bock is an excellent supplement to the ordinary analytical treatises, and covers a wide range of elements.

L. ARCHBUTT.

FOOD INSPECTION AND ANALYSIS. By ALBERT E. LEACH. Revised and enlarged by Andrew L. Winton. New York: John Wiley and Sons; London: Chapman and Hall. 1913. Price £1 11s. 6d.

The recent death of the author of this work is doubtless responsible for the statement on the title-page that the revision of this third edition has been undertaken by Dr. A. L. Winton. In his prefatory remarks the reviser points out that new matter equivalent to about eighty pages has been added, and that much antiquated matter has been replaced by new. When a reviewer has dealt with an early edition of a certain work, it is only natural that when criticising a succeeding edition he should first turn to those portions on which he had previously felt himself compelled to pass more or less adverse criticism. In the present instance this proceeding has been productive of disappointment, for very few of the errors of omission or commission to which attention was called in the previous review appear to have been rectified.

In dealing with the detection and estimation of arsenic, no reference is made to the work of the Joint Committee of the Society of Chemical Industry and the Society of Public Analysts, and the author seems to have been unaware that it was the labours of that body which first rendered possible the accurate determination of minute traces of arsenic in food-stuffs. Analysts are instructed in carrying out the "Marsh test" to add "20 to 30 grms. of arsenic-free stick zinc and a perforated 'platinum' disk to form an electric couple." The author appears to have overlooked all the work that has been done on the insensitiveness of zinc, and if American analysts generally are accustomed to follow the procedure recommended in this work, it would be a matter for some surprise if they were to find more than "insignificant traces" of arsenic in the food-stuffs which they are accustomed to examine. The quite unnecessary destruction of organic matter in the case of beer and certain other liquids is recommended, and the operator (working on 100 c.c. of beer) is told that the "arsenic mirror may be weighed in the usual manner if of sufficient size." The extreme insensitiveness of the test as carried out under the conditions mentioned in the text is exemplified by the fact that one of the mirrors intended for comparison purposes is made with as much as $\frac{1}{20}$ mgrm. of arsenious oxide (p. 76), and it is directed that the test should run for from two to three hours. In good practice and with a sensitive apparatus no mirror corresponding with more than $\frac{1}{100}$ mgrm. can safely be used. Reference is made to the cases of arsenical poisoning by contaminated beer which occurred in this country in 1900, but the possibility of heavy traces of arsenic being introduced through the medium of carelessly dried malt has been quite overlooked. The above will serve to show how inadequate has been the treatment of this particular subject, and it is to be hoped that before another edition appears the reviser will take steps to familiarise himself with the work which has been done in this country on the estimation of traces of arsenic and on the bearing of the question of electrical supertension on the sensitiveness of the test. While speaking of beer, the reviewer cannot refrain from calling attention to the somewhat extraordinary statement that the lack of gas caused by the absence of proper cask fermentation is "sometimes made up by the addition of sodium bicarbonate."

In the section dealing with metallic impurities in tinned food, no mention is made of the influence of air (oxygen) in the tins, as affecting the solution of the metal, although attention is directed to a number of much less important factors. Not yet does the valuable report on this subject of the Local Government Board published in 1908 appear to have found its way into the Chicago Food and Drug Inspection Laboratory, and not yet does Schryver's colorimetric method receive any mention.

"Malt Extract" would appear, in the author's view, to be a designation restricted to a material prepared according to certain directions contained in the United States Pharmacopœia, 1880. We are told that it "has a residue of at least 70 per cent.," and consists "chiefly of maltose, and contains *about* 2 *per cent. of diastase*" (the italics are the reviewer's). In a work devoted to analytical chemistry we are actually supplied with three analyses of pure malt extract, in not one of which is any dextrose shown.

In the section on butter analysis, the author, when discussing the Reichert-Meissl number, repeats the statement of the earlier edition that "the result is conclusive, excepting in those rare instances when the admixture of foreign fat is so small as to cause the Reichert number to approximate that of pure butter." No mention is made here (and very little elsewhere) of the effect of the presence of cocoanut oil, and no more unsuitable word than "conclusive" could have been used in the above connection.

The chapter on sugars and saccharine products is on the whole good, but it is worthy of remark that no reference is made in the text to any of the work of Horace Brown and others in this country, and we are informed that "maltose is of little importance from the standpoint of the food analyst excepting as an ingredient of commercial glucose, and as being the sugar produced by the action of ptyaline . . . on the starch of food in the ordinary process of digestion." Having regard to the quite correct statement on p. 356 that the sugars of a considerable number of infants' and invalids' foods consist largely of maltose, this needs some modification. It is not correct, moreover, to say that few complete analyses of infants' foods have been made, and much more complete series than that given have been published.

In the section dealing with the estimation of chicory in coffee, the average specific gravities of coffee and chicory infusions are given to five decimal places, but no warning is conveyed to the analyst as to the extent to which the percentage of extract in chicory may vary, depending on the degree of roasting and other factors.

Under the heading "Vegetable and Fruit Products," ketchup occupies an amount of space which would appear to be out of all proportion to its importance. In connection with the examination of this condiment, in a paragraph headed "Estimation of Yeasts and Spores" the following remarks occur:

"These are counted together because of the difficulty in differentiation without making cultures, which is impossible with a sterilised product."

The analyst is then instructed to shake a certain quantity of the material with some water, to examine in a Thoma-Zeiss cell with a magnification of 180, and to "count the number of yeasts and spores in one-half of the ruled squares, which gives the number present in $\frac{1}{60}$ of a cubic millimeter of the original material." The first sentence is unintelligible. If the yeast cells were dead, they would necessarily have undergone disintegration, and it is doubtful if they could be identified with a magnification of 180 diameters; whereas if they were living, there should be no difficulty in making cultures, assuming such a proceeding to be desirable. The further statement in reference to yeast cells and mould spores, "the number in home-made and best factory-made ketchups is practically none; the allowed limit is 25," is not devoid of humour.

Apart from the transatlantic phonetic spelling, to which we are now becoming accustomed, and which has in some instances (*e.g.*, levulose) sacrificed all considerations of derivation to what is regarded as practical utility, there are a good many misprints and occasional inelegancies of diction. Thus we are told that port wine on aging "looses considerable of its astringency," and quite a number of proper names are misspelt, notwithstanding that attention was called to these in the review of the preceding edition. Bourguelot still appears for Bourquelot, Sangle-Ferriere for Senglé-Ferrière, Bechi for Becchi, Villivecchia for Villavecchia, Blythien for Beythien, Klocker for Klöcker, and Plummer for Plimmer. Some of the photomicrographic illustrations at the end of the book are good, but others are so indistinct as to have little or no practical value.

The above criticisms are made in no ungenerous or carping spirit. The book is much too good to become solely a textbook for the American analyst and food inspector, and is well qualified to take an international position as a work of reference on the analysis of food-stuffs. One of its main drawbacks, however, and one which must limit materially its sphere of usefulness, is the apparent impression of both author and reviser (notwithstanding numerous bibliographical references to foreign journals) that food chemistry is an almost exclusively American branch of science. It would not be good taste on the part of the reviewer to point out how far that is from being the case, but he may perhaps be permitted to express the hope that when future editions are in course of preparation, Dr. Winton or some other reviser may see his way to study a little more closely the literature of other countries, and to free himself rather more from the bondage of United States Departmental Reports and Official Bulletins. In this way only can this work take the full position to which its many merits entitle it.

A. Chaston Chapman.

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OBITUARY.

JULIUS LEWKOWITSCH.

CHEMISTRY has suffered a very great loss in the death of Julius Lewkowitsch, which occurred at Chamonix on September 15, after only a few days' illness. A technologist of outstanding merit, the recognised authority on vegetable and animal fats and oils, an author of marked originality and reliability, and an energetic and stimulating member of the councils of many societies associated with both pure and applied chemistry, his work will go down to posterity as a permanent tribute to a life of exceptional and successful activity. .

Lewkowitsch was born at Ostrowo, in Prussian Silesia, in 1857, and received his vocational training, under Victor von Richter, at the University of Breslau, where he took the degree of Ph.D. in 1879, and that of M.A. in 1881. With the intention of devoting himself to an academic career, he accepted a position as teacher of mathematics and natural science at the Friedrich's Real Gymnasium, Berlin, under Landolt, and subsequently became assistant to Victor Meyer at Heidelberg University. A few years later, however, he turned from the promising career that appeared to be opening out to him in pure science, to what subsequently proved to be his life's work in chemical technology. After occupying the positions of research chemist at a tar and petroleum works, and manager of a tar distillery in Germany, he came to England, when about thirty years of age, to act as chemist and technical manager to Messrs. Watson and Sons at their Leeds soap works. This last post he held for some eight years, during which he became naturalised as a British subject. In 1895 he commenced his work as a consultant in Manchester, and in 1898 transferred his rapidly extending practice to London.

Lewkowitsch's contributions to chemistry are associated respectively with the two separate periods of his career, as a teacher and as a technologist. During the comparatively short time that he was engaged in academic work, he published several notable papers. His first contribution was a study of the action of nitric acid on fatty acids, but in 1882 and 1883, whilst working with Landolt, he turned his attention to the experimental study of stereochemistry, a subject which was then quite in its infancy, and successfully developed Pasteur's method for the resolution of externally compensated substances by means of living organisms. By the action of *Aspergillus mucor* and of *Penicillium glaucum* on the racemic form of ammonium mandelate, prepared from benzaldehyde, he obtained the dextro-rotatory stereoisomer of mandelic acid, and subsequently prepared the optically active modifications of lactic, glyceric and tartaric acid by similar means. From these investigations he was led to the study of the possible asymmetry of benzenoid compounds, from the consideration that, according to Ladenburg's prism formula for benzene, an orthodisubstitution product should be optically active, and attempted to isolate the optically active forms of the two methyl salicylic acids and of other compounds of similar orientation.

In the field of technology, to which Lewkowitsch devoted the greater part of his life, his work was concentrated on the subject he made so particularly his own, and on which it is not too much to say he was the greatest living authority—the technology of fats and oils. To this journal he contributed papers on the estimation of glycerol; on the separation of oleïc acid from other fatty acids; on the differentiation of linseed oil from boiled oils; on oxidised oils, Indian oils, Carapa oil, and on a variety of the less known fats. To the Society of Chemical Industry he contributed the results of many important investigations bearing on analytical problems connected with the oil, fat and wax industries, in addition to papers on subjects of a wider scope, such as the "Theory of Saponification" and "Problems in the Fat Industry." These contributions, together with many others to foreign journals, are for the most part embodied in his standard book on the "Chemical Technology and Analysis of Oils, Fats and Waxes." This work, as first published in 1895, was founded on Benedikt's "Analyse der Fette und Wachsarten," to which Lewkowitsch made considerable additions; the second edition, which followed in 1898, was practically an independent work, which was much expanded and brought up to date in the subsequent third and fourth editions, which were published in 1904 and 1909 respectively. A fifth edition has been issued during the present year, of which the first volume was published in July and the second volume is in the press. In addition to the English edition of his book, Lewkowitsch also published separate editions in both French and German. His well-known "Laboratory Companion" to this work was published in 1901. His numerous other literary contributions include a course of Cantor Lectures before the Royal Society of Arts in 1904, on "The Uses and Application of Oils and Fats," and the articles on oils and fats in Thorpe's "Dictionary of Applied Chemistry," in Lunge's "Chemisch-technische Untersuchungsmethoden," and in the English edition of the same work, "Technical Methods of Chemical Analysis," for which he had also revised the sections on mineral oils, lubricants, and resins and balsams; he also wrote the sections on oils and fats in the "Encyclopædia Britannica," and in Meyer's "Jahrbuch der Chemie."

The preface to Lewkowitsch's last published work, the first volume of the fifth edition of his "Chemical Technology and Analysis of Oils, Fats, and Waxes" includes this statement: "Whereas a decade or two ago chemical analysis pointed out the way to technical development, and purely scientific discoveries appeared to have comparatively little influence on the progress of our industries, the order is now reversed. In its influence on the advance of the oils and fats industries pure science has stepped ahead, and it is now the turn of analytical chemistry to follow in the wake of progress and to detect in the finished articles the achievements of technical work." Lewkowitsch's own work is a striking example of the manner in which analytical chemistry should "follow in the wake of progress." Few men recognised more fully the value of the services rendered by analytical chemistry to the advance of technology, and the fundamental importance of analytical accuracy in the control of the industrial applications of chemistry.

The services rendered by Lewkowitsch to scientific societies were always marked by helpful and wise counsel. He served on the Council of this Society in 1902-3, on that of the Chemical Society during the same period, on that of the Institute of Chemistry from 1901 to 1904, and he was very specially associated with the work of the Society of Chemical Industry, of which he was the Foreign Secretary at the time of his death. In 1909 he was awarded the Lavoisier Medal as Conférencier of the Société Chimique de France.

Apart from his great scientific attainments, Lewkowitsch was a man of marked individuality, with wide sympathies and knowledge, ever ready to help his fellow-workers, especially younger men, many of whom owe their success to his guidance and training. He gave his services ungrudgingly to whatever he undertook, with all too little regard to his own leisure and recreation; and although he was recognised as a strong advocate and as a powerful adversary, he has left none but the best of friends amongst a large circle, which, owing to his exceptional linguistic abilities, extended far beyond the confines of his adopted country.

Apparently in the full vigour of life, and with every promise of many further years of activity, Lewkowitsch passed away within sight of the Alps he knew so well, and on which he enjoyed his chief recreation. He married in 1902, and is survived by his wife and a young son and daughter.

CHARLES A. KEANE.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Nitrogenous Substances in Barley and Malt. III.: Transformation of the Proteins during Mashing, Boiling of Wort, and Fermentation. H. Schjerning. (*Compt. rend. des Travaux du Lab. de Carlsberg*, 1913, **9**, 237-396.)—The analytical methods employed are similar to those described in a former paper (ANALYST, 1910, **35**, 350). The acidity of wort is now determined by a titration with $\frac{N}{10}$ sodium hydroxide "to the tone of colour which is produced when 2 c.c. of a solution of phenolphthaleïn (1 per cent.) and 4 c.c. of a $\frac{N}{15}$ solution of disodium hydrogen phosphate are added to 25 c.c. of distilled water which has previously received an addition of Bismarck brown and tropeolin O in quantity to give the solution the same colour as the wort the acidity of which is to be determined." Ammoniacal nitrogen is estimated by distilling the wort with a convenient excess of magnesium oxide. The amino-acid nitrogen titratable with formaldehyde and the concentration of hydrogen ions were determined in a dilute wort by Sörensen's method (ANALYST, 1908, **33**, 19; 1909, **34**, 498). Peroxidase and catalase were measured by methods previously described (*Compt. rend. des Travaux du Lab. de Carlsberg*, 1909, **8**, 67; 1910, **8**, 200, 321; ANALYST, 1910, **35**, 350). A large amount of experimental matter is included in the paper, together with over fifty tables of figures, the summary of the "principal results" alone occupying ten pages of matter. Through all the stages of mashing, boiling, cooling, fermentation (primary and secondary), the effect of different water treatment, wort concentration, mashing temperatures, boiling-time, hop variation, aëration, etc., on the wort is followed by estimating, among other variables, the amounts of total extract, ash, acidity, total nitrogen, assimilable nitrogen, peptic cleavage products, tryptic cleavage products, ammonia, "albumin I. and II.," proteose, peptone, denuclein, and hydrogen ion concentration.

H. F. E. H.

Composition of the Seeds of Cicer Arietinum, L. (Chick-Pea). A. Zlataroff and S. Stoikoff. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1913, **26**, 242-247.)—The chick-pea is a dwarf pea cultivated in Bulgaria and Southern Europe generally, and used as a food like lentils. Results of analyses of twenty-four samples showed it to have the following average composition: Water, 10·47; proteins, 22·62; fat, 5·08; starch, 49·33; crude fibre, 3·09; ash, 2·88 per cent. The total phosphoric acid amounts to 0·88 per cent (as P_2O_5), of which 0·125 per cent. is present as lecithin-phosphoric acid. The peas are frequently roasted before use, and are then known under the name of "leblebiji." A product known as "seimitt" is prepared by allowing the peas to germinate and ferment. The germinated peas contain oxydase, zymase, protease, diastase, and rennin; and the products of fermentation are hydrogen, hydrogen sulphide, carbon dioxide, butyric acid, lactic acid, succinic acid, sugar, ethyl and higher alcohols, leucine, asparagine, and aromatic substances (aldehydes and esters?). W. P. S.

Examination of Chloroform. K. Enz. (*Apoth. Zeit.*, 1913, **28**, 672-674; through *Chem. Zentralbl.*, 1913, II., 1172-1173.)—The impurities of commercial chloroform include chloral alcoholate and similar chloral compounds. These may be estimated by treating the chloroform with $\frac{N}{10}$ alkali and titrating the excess. Pure chloroform is not affected by alkali of this strength. Aldehydic and similar compounds are detected by means of Nessler's reagent, which gives with impure chloroform a red-brown deposit rapidly changing to greenish black. Pure chloroform does not give any reaction. C. A. M.

Detection of Formaldehyde in Foods. F. Rachel. (*Pharm. Zentralhalle*, **54**, 759-761; through *Chem. Zentralbl.*, 1913, II., 903.)—Friese's method for the detection of formaldehyde in milk has been modified to make it applicable to meat, caviare, and fish preparations. The sample is treated with phosphoric acid and distilled with steam in the usual way. One to two c.c. of the distillate are thoroughly shaken with 4 c.c. of milk free from formaldehyde and 10 c.c. of hydrochloric acid (sp. gr. 1·19) containing 1 drop of 25 per cent. nitric acid in 300 c.c. If formaldehyde be present, a blue coloration is immediately produced. Since the reaction only takes place with traces of formaldehyde, it is advisable, in the case of a negative result, to repeat the test after diluting the distillate. In order to determine whether the formaldehyde is present in the free or combined condition, a separate portion may be distilled without the addition of phosphoric acid. J. F. B.

Estimation of Total Formaldehyde in Fumigators and Commercial Solutions. J. J. Hinman jun. (*J. Ind. and Eng. Chem.*, 1913, **5**, 752-755.)—The author has tested comparatively, by nine different methods, a number of samples of formaldehyde preparations containing polymerides and foreign matters. The ammonia methods are not very rapid, and the end-point of the titration in presence of rosolic acid is not distinct. The litmus end-point is more satisfactory. The hydrogen peroxide methods are more rapid, and the results are easily checked. The end-point of the sulphite methods is sharper than in either of the other processes. The titrations are direct. For formaldehyde solutions they require the minimum of time, but when polymers are to be titrated the Kleber method is the most rapid of all. For an occasional analysis the peroxide method of Haywood and Smith (ANALYST, 1905, **30**, 401), using litmus indicator, or the sodium sulphite method of Seyewetz and Gibello (ANALYST, 1904, **29**, 288) seem to be preferable, whilst for regular analyses of large numbers of samples the bisulphite method of Kleber (ANALYST, 1904, **29**, 161) would be most suitable. Legler's ammonia method gives too low results, and Blank and Finkenbeiner's peroxide method gave the highest results. Haywood and Smith's method gave slightly lower percentages than Blank and Finkenbeiner's, closely followed by Kleber's and the other methods.

J. F. B.

Baobab Oil. V. Thomas and F. Boiry. (*Bull. Soc. Chim.*, 1913, **13**, 827-832.) —The seeds from different varieties of baobab (*Adansonia*) contain considerable quantities of oil, but those of *Adansonia grandidieri*, a tree growing in Madagascar, appear to contain the largest amount. These seeds, in the undecorticated condition, yield 43 per cent. of a yellow-coloured oil, whilst the decorticated seeds yield 65·4 per cent. of a white fat having the consistence of butter. The following figures were obtained on the examination of the oils, and the fatty acids separated from them:

	From Whole Seeds.	From Decorticated Seeds.
Oil:		
Sp. gr.	0·9190 (at 20° C.)	0·9135 (at 40° C.)
Melting-point	20°–21° C.	39°–40° C.
Solidifying-point	13° C.	33° C.
Refractive index at 40° C. ...	1·4585	1·4521
Saponification value	192·4	196·0
Iodine value (Wijs)	65–66	36·9
Reichert-Meissl value ...	0·77	—
Hehner value	95·5	
Fatty Acids:		
Melting-point	51°–52° C.	45°–46° C.
Solidifying-point	44·5° C.	—
Acid value	179·0	204·5
Saponification value	202·5	207·6
Iodine value	66–67	34–35

The difference between the acid and saponification values in the case of the oil from the undecorticated seeds indicates the presence of lactones, and further examination showed that the fatty acids contained 11·4 per cent. of lactones, the nature of which, owing to lack of material, could not be investigated. The mixed fatty acids consisted of myristic acid, 7 per cent.; palmitic acid, 32·5 per cent.; oleic acid, 36·5 per cent.; and linoleic acid, 8·7 per cent. Stearic and arachidic acids were not present.

W. P. S.

Hickory Nuts and Hickory Nut Oil. G. O. Peterson and E. H. S. Bailey. (*J. Ind. and Eng. Chem.*, 1913, **5**, 739-740.)—Two varieties of hickory nuts have been studied—*Carya ovata*, or shell-bark hickory, the edible variety, and *C. amara*, the swamp hickory, the nuts of which are only fed to pigs. The kernels of the edible nuts have a high food value, containing 70·2 per cent. of oil and 13·2 per cent. of protein. By cold pressing, followed by hot pressing, 65 to 66 per cent. of the total oil present may be extracted. The oil retains the flavour of the hickory nut, and is practically equal to olive oil as a table oil. The oils obtained from the two species of nut are practically identical, and are similar to cottonseed oil in their chemical and physical characters. The resemblance is so close that a differentiation from cottonseed oil would be difficult. The following constants were obtained for the two varieties of oil, and are compared with those recorded for a sample prepared at the Maine Experiment Station:

CONSTANTS OF HICKORY NUT OILS.

	C. Amara.	*C. Ovata.*	Maine Experiment Station.
Sp. gr. at 24° C.	—	0·9119	0·9164
Refractive index at 20° C. ...	1·4699	1·4699	1·4696
Hehner value	95·6	95·7	—
Reichert-Meissl value ...	0·48	0·47	—
Iodine value	105·2	106·8	102·8
Saponification value	190·0	189·6	—

J. F. B.

Analysis of Maple Products. I.: Electrical Conductivity Test for the Purity of Maple Syrup. J. F. Snell. (*J. Ind. and Eng. Chem.*, 1913, **5**, 740-747.)—When maple syrup is adulterated with commercially pure sugar, the percentage of non-sugar constituents is reduced, and, since sugar is a non-conductor, the electrical conductivity of the syrup falls. An examination of a number of samples of genuine maple syrups showed that their conductivities varied within somewhat wide limits inversely as their densities. On dilution with water the conductivities increased until a point of maximum conductivity was reached, after which the conductivities decreased owing to the dilution of the electrolytes. The point of maximum conductivity occurs at a dilution corresponding to one volume of syrup to two volumes of water. This observation is made the basis of a rapid test for the purity of maple syrups. A suitable quantity of the syrup (15 c.c.) is measured out in a graduated cylinder and drained into a small beaker; the cylinder is then twice washed out with equal volumes of water, and the syrup is dissolved therein. The solution is poured into the conductivity cell, the temperature is adjusted to 25° C., and the measurement is made. The constant of the cell is divided by the number of ohms, and the result multiplied by 100,000. The values for genuine syrups lie between 110 and 200, but further experience may extend these limits a little. Syrups giving conductivity values distinctly outside these limits may be condemned without further examination; but those giving normal values are not necessarily pure, and should be examined by the usual methods. The essential features of the apparatus are: A low voltage electrical current operating an induction coil; a conductivity cell of a form suitable for liquids of low conductivity and with electrodes not easily displaced; a Wheatstone bridge with telephone; a device for exact regulation of temperature. An examination of the ash of pure maple syrups indicates that the organic salts of potassium and calcium are the principal electrolytes of the syrups, and the tabulated results show close relationships on the one hand between conductivity and total ash, and on the other hand between conductivity and alkalinity of the soluble ash. Estimations of conductivity values of non-maple syrups showed that syrup from granulated sugar is practically a non-conductor, but that molasses show values many times greater than the average for pure maple syrup. Syrup made from pale brown sugar showed a value of 178, thus within the limits for pure maple, and it is evident that various mixtures of partially refined products could be made to show normal values. Consequently a normal conductivity value is in no sense evidence of purity, but the method is extremely rapid and useful for sorting out obviously adulterated samples (*cf.* ANALYST, 1912, **37**, 538, 543).

J. F. B.

Detection of Added Water in Milk by Means of the Refractometer Value of the Serum. G. Schutz and L. Wein. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1913, **26**, 177-184.)—The serums obtained, respectively, by the use of calcium chloride (ANALYST, 1907, **32**, 117) and by means of carbon tetrachloride and acetic acid (*ibid.*, 1912, **37**, 450) are equally suitable for refractometric purposes. The calcium chloride method of preparing the serum is simple and rapid, whilst the carbon tetrachloride method yields a serum which may be used for determining constants other than the refractometer value. In the case of the calcium chloride serum, the increase in the refractometer value, due to the formation of acidity in the milk, may be prevented by the addition of a few drops of formaldehyde to the milk. Another source of error consists in the solubility of precipitated lime compounds when the serum is allowed to remain too long a time in contact with the coagulum; the serum should be separated within thirty minutes. W. P. S.

Black Mustard Powder. H. Imbert and A. Juillet. (*Bull. Sciences Pharmacol.*, 1913, **20**, 385-388; through *Chem. Zentralbl.*, 1913, II., 1170-1171.)—Allyl mustard oil is estimated by the Codex method of hydrolysing the potassium myronate by means of myrosin in the presence of water. For the detection of white mustard, 5 grms. of the powder are boiled for two minutes with 100 c.c. of water containing 20 drops of alkali hydroxide solution, and the boiling mixture diluted with 200 to 300 c.c. of cold water. The deposit is separated, washed two or three times, and examined under the microscope for uncrushed particles of white mustard seed. Black mustard powder of different origin was found to contain from 0·493 to 0·899 per cent. of mustard oil, whilst a sample of white mustard powder contained 0·068 per cent. Many samples of the black powder contain turmeric and linseed-meal as impurities. C. A. M.

Composition of Rums. J. Sanarens. (*Ann. Falsif.*, 1913, **6**, 488-495.)—Analyses of numerous authenticated samples of rum are recorded, and the minimum and maximum results obtained are given in the following table; the results obtained on the examination of six samples of artificial rum are also given. The figures express grms. per 100 litres of absolute alcohol.

	Rum imported from—						Artificial Rum.
	Martinique.	Guadeloupe.	Réunion.	Cochin-China.	British Guiana.	Jamaica.	
Total acids:							
Minimum ...	65	82	128	20	80	123	82
Maximum ...	394	138	245	50	87	176	159
Aldehydes:							
Minimum ...	3	5	4	6	10	13	4
Maximum ...	90	36	41	17	41	16	14
Esters:							
Minimum ...	37	31	56	12	24	632	127
Maximum ...	797	72	101	29	46	938	2,240
Higher alcohols:							
Minimum ...	19	80	73	153	139	22	14
Maximum ...	295	194	93	319	164	39	70
Furfural:							
Minimum ...	1	1	1	trace	1	1	1
Maximum ...	9	3	4	1	2	2	5

W. P. S.

Examination of Turpentine Oil by the Methods of the German Pharmacopœia. H. Wolff. (*Pharm. Zeit.*, 1913, **58**, 470-471; through *Chem. Zentralbl.*, 1913, II., 1085-1086.)—For the estimation of the bromine value, 2 c.c. or 2 grms. of the sample are made up to 100 c.c. with petroleum spirit, and 4 c.c. of the solution added to a mixture of 50 c.c. each of the potassium bromate and potassium bromide solutions of the German Pharmacopœia. Five c.c. of sulphuric acid are then added and the flask immediately stoppered, and shaken for about a minute, after which the turpentine solution is added drop by drop with continual shaking, until the petroleum spirit layer is decolorised. The bromine value is calculated from the total number of c.c. of the turpentine solution consumed. If 2 grms. of the sample were taken the value is $\frac{1,200}{x}$; and if 2 c.c. were taken it is $\frac{1,200}{xs}$, where s represents the sp. gr. of the turpentine oil. Pure oil of turpentine has a bromine value of 215 to 230, rarely as low as 210.

Test for Pine Oil.—Five c.c. of the sample are boiled with 5 drops of nitrobenzene, 2 c.c. of 25 per cent. hydrochloric acid then added, and the liquid again boiled for ten seconds. All pine oils give a deep brown coloration of the oily layer, whilst the acid layer becomes brown or black. In the case of turpentine oil there is either no coloration, or the oily layer shows a greenish tint, whilst the acid layer is coloured light brown. By means of this test an addition of 5 per cent. of pine oil to oil of turpentine may be detected. C. A. M.

Estimation of Chlorides in Wine. C. von der Heide and M. Kartschmar. (*Zeitsch. anal. Chem.*, 1913, **52**, 645-651.)—In wines containing less chloride than corresponds to 5 mgrms. chlorine per 100 c.c., it is unnecessary to add alkali before igniting the residue; but with wines containing 10 mgrms. or more chlorine per 100 c.c. there is considerable loss if the addition of alkali be omitted. Incinerating wine residues to which alkali has been added is troublesome, often attended with loss, and the authors have tried several alternative methods for the estimation of chlorine in wine, and recommend the following, which gives exact results with natural wines: A measured and more than sufficient amount of $\frac{N}{50}$ silver nitrate solution is added to 100 c.c. of wine, together with 2 to 3 grms. of animal charcoal in the case of white wines, or 5 to 8 grms. in the case of red wines. The mixture is boiled for fifteen minutes, 20 c.c. of dilute nitric acid is then added, boiling continued for a further ten minutes, and the solution filtered. The charcoal and filter are washed with 200 to 300 c.c. of hot very dilute nitric acid, and the filtrate and washings are allowed to cool. Carbamide is added if necessary to destroy nitric oxide, and the excess of silver titrated with thiocyanate. Direct volumetric determination of chlorine in the original wine is impossible on account of the colour, and the results of gravimetric determinations on the original wine are always too high.

Musts and very sweet wines are best treated as follows: The sugar is fermented away as far as possible, and the fermented liquid is evaporated to a syrup, mixed with 10 grms. of slaked lime, dried, and burnt. The ash is extracted with water, and the aqueous extracts are filtered into a vessel containing 25 or 50 c.c. $\frac{N}{10}$ silver nitrate solution. Finally the lime is dissolved in dilute nitric acid, and this solution also filtered (from particles of carbon, etc.) into the vessel containing the silver nitrate. The excess of silver is then determined by titration with thiocyanate.

G. C. J.

Estimation of Tartaric, Succinic, and Malic Acids in Wine. P. Dutoit and M. Duboux. (*Bull. Soc. Chim.*, 1913, **13**, 832-862.)—A detailed description is given of the combined volumetric and conductivity method (*cf.* ANALYST, 1909, **34**, 56; 1912, **37**, 600) for the estimation of organic acids in wine. The procedure is as follows: The total acidity and sulphates are first estimated, and let *a* denote the number of c.c. of $\frac{N}{1}$ alkali solution required to neutralise 1 litre of the wine. One hundred c.c. of the wine are then heated in a flask to boiling, and a quantity of barium chloride solution just sufficient to precipitate the sulphates is added, followed by the addition of 2 c.c. of a 25·2 per cent. uranium nitrate solution. The mixture is shaken for a few minutes, neutralised with sodium hydroxide, 0·18 c.c. × *a* of $\frac{N}{1}$ silver nitrate solution and 250 c.c. of 95 per cent. alcohol are added, and the mixture is filtered. The filter and precipitate are transferred to the flask, and mixed thoroughly with 0·12 c.c. × *a* of $\frac{N}{1}$ sodium bromide solution (prepared with 50 per cent. alcohol) and 100 c.c. of 47·5 per cent. alcohol. After the lapse of about ten minutes the mixture is diluted to 202 c.c. with 47·5 per cent. alcohol, and filtered. The filtrate now contains the organic acids, and is used for the estimation.

Estimation of Tartaric, Malic, and Succinic Acids.—A definite volume of the solution of the acids (according to the acidity of the wine; see below) is placed in the conductivity vessel, an equal volume of 47·5 per cent. alcohol is added, then 0·5 c.c. of $\frac{N}{10}$ acetic acid, and the conductivity of the solution is determined while the latter is titrated with $\frac{N}{2}$ lanthanum nitrate solution which has been standardised in a similar way against $\frac{N}{2}$ sodium tartrate solution.

Acidity of the Wine in c.c. $\frac{N}{1}$ per Litre.	Volume of the Acid Solution to be taken.	Volume of 47·5 per Cent. Alcohol to be added.
Below 90	25 c.c.	25 c.c.
90–115	20 „	30 „
115–140	15 „	35 „
Above 140	10 „	40 „

Estimation of Tartaric, and Malic Acids.—Another quantity of the solution (see below) is treated with 1 c.c. of glacial acetic acid and alcohol, and titrated with $\frac{N}{2}$ lanthanum nitrate solution while the conductivity is determined. In this and in the previous titration the lanthanum nitrate solution is added in quantities of about 0·1 c.c. at a time:

Acidity of the Wine in c.c. $\frac{N}{1}$ per Litre.	Volume of the Acid Solution to be taken.	Volume of 47·5 per Cent. Alcohol to be added.	Volume of 76 per Cent. Alcohol to be added.
Below 90	25 c.c.	0 c.c.	25 c.c.
90–115	20 „	5 „	25 „
115–140	15 „	10 „	25 „
Above 140	10 „	15 „	25 „

Estimation of the Tartaric Acid.—Two titrations may be necessary, according to the amount of malic acid also present. A portion of the solution containing the acids is mixed with 5 c.c. of glacial acetic acid and 75 c.c. of 95 per cent. alcohol, and the conductivity is determined while the mixture is titrated with barium acetate solution, the barium equivalent of which has been ascertained by the conductivity method. If from the results obtained the ratio of malic acid to tartaric acid is less than 1 : 3·7, a second titration must be made, as the presence of much malic acid renders the first titration untrustworthy. Another portion of the solution is treated with $\frac{N}{2}$ sodium tartrate solution, the quantity added being $\frac{3\cdot7\,m - t}{1{,}000}$ c.c. per c.c. of the acid solution taken, m expressing the amount of malic acid per litre in c.c. of $\frac{N}{1}$ solution, and t the amount of tartaric acid in c.c. of $\frac{N}{1}$ solution. Five c.c. of glacial acetic acid and 75 c.c. of 95 per cent. alcohol are then added, and the mixture is titrated with the barium acetate solution.

Sum of Tartaric + Malic + Succinic Acids in c.c. $\frac{N}{1}$ per Litre.	First Titration.		Second Titration.	
	Volume of Acid Solution to be taken.	Volume of 47·5 per Cent. Alcohol to be added.	Volume of Acid Solution to be taken.	Volume of 47·5 per Cent. Alcohol to be added.
Below 70	25 c.c.	0 c.c.	20 c.c.	5 c.c.
70–90	20 „	5 „	15 „	10 „
90–110	15 „	10 „	10 „	15 „
110–130	10 „	15 „	5 „	20 „

When excessively large quantities of malic acid are present in wines, it is advisable to estimate the tartaric acid separately by the ordinary potassium hydrogen tartrate method. This also applies to wines containing citric acid, as this acid is estimated together with the tartaric acid in the conductivity-titration method. W. P. S.

Estimation of Tartaric, Citric, Lactic, and Malic Acids in Musts, Wines, etc. L. Mathieu and L. Ferre. (*Bull. Assoc. Chim. Sucr. et Dist.*, 1913, **30**, 842-844; through *Chem. Zentralbl.*, 1913, II., 709.)—In the case of wines the acids are first separated by Mestrezat's method (Analyst, 1907, **32**, 266, 388), and the lactic acid in the filtrate estimated by Möslinger's method. The tartaric acid is estimated in a portion of the precipitate, dissolved in sulphuric acid, by the method of Kling (Analyst, 1912, **37**, 516), whilst another portion of the precipitate is oxidised by Mestrezat's method in acid and in alkaline solution. One grm. of tartaric acid is oxidised by 1·447 grms. of potassium permanganate in acid solution, and by 0·620 grm. in alkaline solution; 1 grm. of malic acid by 2·078 grms. in acid solution, and 0·615 grm. in alkaline solution; and 1 grm. of citric acid by 1·820 grms. in acid solution and 0·480 grm. in alkaline solution. If x grms. of tartaric acid, y grms. of malic acid, and z grms. of citric acid are present, the proportion of y and z may be calculated by means of the following equation, in which V represents the volume of acid permanganate solution, and V^1 the volume of alkaline permanganate solution, and a the strength of the solution. The value of x has been found by direct estimation:

$$y \times 2{\cdot}078 + z\ 1{\cdot}820 = Va - x\ 1{\cdot}447$$
$$y \times 0{\cdot}615 + z\ 0{\cdot}480 = V^1a - x\ 0{\cdot}620$$

The method is applicable even in the presence of succinic acid, since the latter, although precipitated by Mestrezat's method, is not attacked by the permanganate solution, and may be separated by the usual methods (*cf.* Analyst, 1913, 381).

C. A. M.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Methods of Estimation of Carbohydrates, especially in Plant Extracts. W. A. Davis and A. J. Daish. (*J. Agric. Sci.*, 1913, **5**, 437-468.)—The alcoholic extract is evaporated *in vacuo* to a small definite volume, usually 500 c.c. Two portions of 20 c.c. each are dried down *in vacuo* for eighteen hours at 100° C.; this gives the total dry matter in the extract. Another 440 c.c. are treated with basic lead acetate, filtered under pressure on a Büchner funnel and washed to a known volume (2,000 c.c.). This is solution A; 300 c.c. of A are freed from lead by solid sodium carbonate and made up to 500 c.c. This is solution B. (1) 25 c.c. of B are used for direct reduction with Fehling's solution, and is also polarised. The reduction is due to maltose, dextrose, lævulose, and pentoses. (2) For *cane sugar*, 50 c.c. of B are inverted (*a*) by invertase, after making faintly acid to methyl orange with sulphuric acid. One or 2 c.c. of autolysed yeast and a few drops of toluene are added, and the whole left for twenty-four hours at 38-40° C., after which 5 or 10 c.c. of alumina are added and the solution filtered, washed, and made up to 100 c.c. The reducing power of 50 c.c. (=25 c.c. B) is taken, and the solution again polarised. (*b*) By citric acid. The solution is made just acid as before, and a weighed quantity of citric acid is added so as to have 10 per cent. of the crystalline acid present. After boiling ten minutes, cooling, neutralising with caustic soda, the solution is made up to 100 c.c. and treated as before. The cane sugar is calculated from the increase of reducing power and change of rotation, and the values obtained by each of the two methods, (*a*) and (*b*) should agree closely. (3) *Maltose.* Another 300 c.c. of solution A are freed from lead by means of sulphuretted hydrogen and filtered, the precipitate being washed until the total volume of filtrate and washings is about 450 c.c. Air is aspirated through this for about one and a half hours to expel hydrogen sulphide. A little ferric hydroxide is added to remove the last trace of the latter, and the solution is made up to 500 c.c. After filtration, 50 c.c. portions are fermented with (*a*) *S. marxianus;* (*b*) *S. anomalus;* (*c*) *S. exiguus;* (*d*) and (*e*) baker's yeast. Pure culture inoculations from agar yeast-water are employed, and 5 c.c. of sterile yeast-water are added to each flask, the fermentation of the sterile sugar solution being carried on for three to four weeks at 25° C. It is often necessary in order to insure good growth of the yeast first to reduce the acidity by adding 2 to 5 c.c. of $\frac{N}{1}$ sodium carbonate to the 50 c.c. before fermentation. At the end of the fermentation, 5 c.c. of alumina cream are added, the solution made up to 100 c.c., filtered, and 50 c.c. used for reduction. The difference between the average reduction with (*a*) (*b*) (*c*), and the average of (*d*) and (*e*) is the reduction due to maltose. (4) *Pentoses.* These are approximately determined in 50 c.c. of A by distilling with hydrochloric acid according to the A. O. A. C. method, and weighing as phloroglucide. (5) When the reduction in (1) due to pentose and maltose has been allowed for, the remaining direct reduction is due to dextrose and lævulose, the actual proportions of these being calculated from the reducing power and specific rotation. The cupric reduction methods employed throughout are those of Brown, Morris, and Millar (*Trans. Roy. Soc.*, 1897, **71**, 72-123), whose tables are employed. Special attention is drawn to the necessity of boiling the asbestos employed with 20 per cent. caustic soda before use; otherwise with some specimens large errors may be introduced due to the solubility of some of the silicate in the hot Fehling's solution. The copper should never be weighed as Cu_2O, but always as CuO. The volumetric methods of Ling, Rendle, and Jones (ANALYST, 1905, **30**, 182, *ibid.*, 1908, **32**, 160) and Bertrand (*Bull. Soc. Chim.*, 1906, **35**, 1285) have been studied and compared, the former being in all respects preferable to the latter, which can only be regarded as roughly approximate. In fact, the authors find that the volumetric method of Ling is capable of an accuracy of 0·3 per cent. against the 0·3 to 0·5 per cent. found with the gravimetric method. In dealing with plant extracts which have been cleared with basic lead acetate, inversion by citric acid of less than 10 per cent. concentration is incomplete owing to the accumulation of sodium acetate in the solutions analysed. Inversion by invertase is not interfered with by this salt, and both methods should be employed as a check. No loss of sugar occurs owing to the employment of basic lead acetate. It is shown by a detailed study of the action of dilute hydrochloric acid on different sugars that it is impossible completely to hydrolyse maltose at either 70° or 100° C. without destroying simultaneously large amounts of any lævulose or dextrose present. Novelty is claimed for the estimation of maltose in presence of other sugars by means of maltase-free yeasts as above described (*cf.* Baker and Dick, ANALYST, 1905, **30**, 79).

H. F. E. H.

Note by Abstractor.—Three and a half pages of this paper are devoted to the discussion of the fact (well known to sugar chemists) that certain kinds of asbestos used for filtration purposes are to some extent soluble in Fehling's solution unless previously boiled with caustic soda (see Allen, 4th ed., vol. i., p. 335; and Leach, *Food Inspection and Analysis*, 1909, pp. 594, 598). Any loss of this kind would of course become apparent immediately the copper oxide is dissolved and the Soxhlet tube or Gooch crucible weighed again for another determination.

Method of Sensitising Mylius's Reaction for the Detection of Cholalic Acid and Other Biliary Acids. J. Ville. (*Bull. Soc. Chim.*, 1913, **13**, 866-868).—Mylius's reaction depends on the formation of blue, needle-like crystals of the potassium salt of iodocholalic acid when an alcoholic solution of the acid is treated with iodine dissolved in potassium iodide. The reaction is not very sensitive, and the crystals are not obtained when the iodine solution employed is freshly prepared. The author finds, however, that the addition of sodium chloride makes the test an extremely sensitive one, and he recommends the following procedure: The iodine reagent used is prepared by diluting 0·5 c.c. of $\frac{N}{1}$ iodine solution with 200 c.c. of a 33 per cent. sodium chloride solution; if 1 c.c. of this reagent be added to 0·1 c.c. of a 0·2 per cent. alcoholic cholalic acid solution (*i.e.*, 0·0002 grm. of the acid), a blue crystalline precipitate is formed immediately.

W. P. S.

Enzymes of the Tobacco Plant. J. du P. Oosthuizen and O. M. Shedd. (*J. Amer. Chem. Soc.*, 1913, **35**, 1289-1309.)—Loew (*Reports Nos.* 59 *and* 65, *Div. of Veg. Physiology and Pathology, U.S. Dept. of Agric.*) has shown that the fermentation, curing and aroma of tobacco are not due to bacteria (the fermenting leaves being destructive to bacterial life), but are caused by the action of the plant's own enzymes. The following enzymes were tested for in various portions of the plant at different ages:—*Invertase:* Fehling's solution was employed qualitatively to test if invert sugar were formed by the action of the decanted plant extract on cane sugar. *Diastase:* One per cent. starch paste solution was employed, and tested both by iodine and Fehling's solution as above, after standing for sixteen hours at 37° C. in presence of toluene. *Lipase:* Ethyl butyrate and olive oil were employed as substrates, digestions being carried out for various periods of time at 37° C., the solutions being then titrated with $\frac{N}{20}$ caustic potash, using phenolphthaleïn as indicator. *Oxidases:* Two reagents were employed in this test, guaiacum (fresh 20 per cent. tincture previously digested for several days in alcohol) and phenolphthaleïn, both of which were first shown to be reliable in presence of amounts of nitrites in excess of anything that would be found in plants. *Emulsin:* Amygdalin was employed in the tests for this enzyme, after which reducing sugar was tested for with Fehling's solution, the smell of benzaldehyde noted, and hydrocyanic acid tested for. *Inulase:* Inulin solution was employed, and reducing sugar tested for as before. *Proteolytic enzymes* were looked for by the method of Meunier as described in Euler's "General Chemistry of the Enzymes" (English translation, 1912, p. 298). This is based on the fact that, since hydrochloric acid combines with protein during digestion, the diminution of free acid expresses the extent of the action; the method did not prove very satisfactory in practice, trouble being experienced with the end-colours of the titrations. *Reductases:* Methylene blue was used. *Conclusions:* Invertase, diastase, emulsin, and reductases, are present in appreciable amounts in the seed and leaf in all stages of growth and after curing. Lipase, inulase, and a proteolytic enzyme, were possibly present in small amounts. All tests for enzymes in the soil were negative, with one or two exceptions. Oxidases were always present in the green leaf, gradually disappearing as maturity approached, and being absent in the cured leaf. Practically all the starch disappears during the early part of the curing process, and sugar is formed; and this soon disappears, probably by oxidation. There is considerable reduction of the protein content of the leaf during ripening, curing, and fermentation, due to the proteases. A small amount of a glucoside was shown to be present, the breaking down of which by the ferment present may be partly responsible for the aroma. The presence of much fat or protein in the leaf during combustion would impair the flavour, and their removal is effected by the lipase and the proteases present. H. F. E. H.

Distinctions between Pepsin and Peptase. R. Wahl. (*J. Ind. and Eng. Chem.*, 1913, 5, 752.)—The animal proteolytic enzyme, pepsin, may readily be prepared and preserved in the dry state, but the vegetable peptase of malt loses most of its activity when it is attempted to reduce malt extracts of high proteolytic strength to a dry form. Pepsin exerts its maximum effect in the presence of a mineral acid, preferably hydrochloric acid, the secretion of which in the animal organism insures the best conditions for the activity of the digestive enzyme. In an analogous manner, the digestive power of malt peptase is greatly enhanced by the presence of lactic acid of bacterial origin. The presence of this acid is likewise insured under natural conditions by the introduction of lactic bacteria to the steep-water on the surface of the raw barley. The optimum temperatures for the two varieties of enzyme differ materially: pepsin acts most rapidly at a temperature about 50° C.; peptase shows a maximum activity at 35° to 40° C. Peptase is rapidly destroyed at 60° C.; pepsin is not. Barley peptase is capable of exerting an action at a temperature as low as 2° C.; pepsin is inactive at so low a temperature. The usual methods for measuring the proteolytic activity of pepsin are not applicable to peptase. The best test for the proteolytic activity of malt extract is the liquefaction of gelatin. Another, almost equally reliable, is the auto-digestion test by estimating the amount of coagulable protein in the malt extract before and after digestion. Malt extracts rich in active peptase are employed for converting the coagulable colloidal proteins of unstable beers into permanently soluble peptonised forms. J. F. B.

Fish Roe as Human Food. J. König and J. Groszfeld. (*Biochem. Zeitsch.*, 1913, **54**, 351-395.)—Fish roe contains flesh bases (xanthine, hypoxanthine, and creatinin), amino acids (taurine, *l*-tyrosine, glycocoll, and thymine), proteins soluble and insoluble in water, and fat. The proteins are rich in sulphur and phosphorus, and do not yield protamines. The fat is characterised by a high lecithin content (up to 59 per cent.), and also contains cholesterin (3·91 to 14·0 per cent.). Caviare and roe contain free acid which increases in quantity until putrefaction sets in. The ash contains a considerable quantity of free acid, which is due to the sulphur and phosphorus being present mainly in organic combination. E. W.

Occurrence of Succinic Acid in Meat Extract and Fresh Meat. H. Einbeck. (*Zeitsch. Physiol. Chem.*, 1913, **87**, 145-158.)—Succinic acid was found in Liebig's meat extract (about 0·3 per cent.), fresh beef (0·133 grm. in 1·8 kgrm.), and in fresh dog's flesh (0·24 grm. in 1·5 kgrm.). There is no evidence that the quantity increases on storing. E. W.

Occurrence of Carnosine, Methylguanidine, and Carnitin, in Horseflesh. J. Smorodinzew. (*Zeitsch. Physiol. Chem.*, 1913, **87**, 12-20.)—Fresh horseflesh was found to contain 1·82 per cent. carnosine, 0·11 to 0·83 per cent. methylguanidine, and 0·17 to 0·2 per cent. carnitin. E. W.

New Indican Reaction. A. Jolles. (*Zeitsch. Physiol. Chem.*, 1913, **87**, 310-312.)—Urine (10 c.c.) is treated with 2 c.c. of 20 per cent. lead acetate solution, filtered, and to the filtrate 0·5 c.c. of 10 per cent. alcoholic thymol solution, 10 c.c. hydrochloric acid containing ferric chloride (Obermayer's reagent), and 4 c.c. chloroform, are added. On shaking, the presence of even minute traces of indican is indicated by a beautiful violet coloration of the chloroform. This reaction is much more sensitive than any indican test hitherto proposed. *p*-xylenol ($CH_3 : CH_3 : OH = 1 : 4 : 5$) and o-xylenol give similar colorations, that formed by the latter compound being stable towards water and alkalis. The residue which remains after evaporating the chloroform solution dissolves in amyl alcohol to a red solution, and in glacial acetic acid to a violet solution. E. W.

ORGANIC ANALYSIS.

Notes on Alcoholometry. A. F. Joseph and W. N. Rae. (*J. Soc. Chem. Ind.*, 1913, **32**, 856-857.)—The paper deals with the estimation of alcohol in countries where condenser water and air temperatures are abnormally high. The usual tables accompanying the Sikes hydrometer are inaccurate to several per cent. when used at high (80° to 100° F.) atmospheric temperatures. It is seldom recognised that, to obtain a sp. gr. result correct to the fifth place of decimals as given in many tables, the temperature must be adjusted to 0·01° C. Experimental distillations were made in which the temperature of the condenser liquid was varied from 60° to 120° F., the percentage of alcohol being calculated from the tables of Osborne, McKelvy, and Pearce (*Bull. Bureau of Standards* **9**, *Sci. Paper* 197; *J. Soc. Chem. Ind.*, 1913, 304). Condensation was found to be equally efficient at various points between these extremes, estimations being made by means of a brass hydrometer, and also with quartz sp. gr. bottles. There was found to be a large error in using the Sikes hydrometer with the ordinary tables as compared with density determinations, but a great improvement is made if the tables compiled by C. H. Bedford are employed in conjunction with Oertling's glass Sikes hydrometer, Bedford's tables always showing the higher alcohol value. H. F. E. H.

Detection and Estimation of Methyl Alcohol. R. Schmiedel. (*Pharm. Zentralhalle*, 1913, **54**, 709-716; through *Chem. Zentralbl.*, 1913, II., 717.)—The method is based upon the oxidation of the methyl alcohol to formic acid, and estimation of the latter. The sample is cooled to 5° C., rendered alkaline with sodium hydroxide, and treated with successive portions of 3 to 5 c.c. of 1 per cent. hydrogen peroxide solution, first at intervals of thirty minutes and subsequently of forty-five minutes to an hour, until no more gas is evolved. The liquid is then allowed to stand for eight hours, and, after destruction of the excess of hydrogen peroxide by means of thiosulphate, it is acidified and distilled in a current of steam, the distillate being collected in a receiver containing boiling water with calcium carbonate in suspension. About 700 c.c. of the distillate are filtered hot, the filtrate evaporated to dryness, and the residue heated for an hour at 125° to 130° C. It is then dissolved in 100 c.c. of water, and the solution twice extracted with 25 c.c. of ether. The aqueous layer is mixed with 2 grms. of crystalline sodium acetate slightly acidified with hydrochloric acid, and heated with 100 c.c. of 5 per cent. mercuric chloride solution for two hours in a boiling water-bath. The precipitated mercurous chloride is washed by decantation with hot water, and then in a Gooch crucible with hot water, alcohol, and ether, and dried until constant in weight. One grm. of mercurous chloride corresponds to 0·0875 grm. of formic acid and 0·0678 grm. of methyl alcohol. C. A. M.

Estimation of Cellulose by Means of Nitric Acid. V. Rao and B. Tollens. (*J. für Landw.*, 1913, **61**, 237-244; through *Chem. Zentralbl.*, 1913, II., 903.)—The authors have simplified the method of Dmochowski and Tollens (ANALYST, 1910, **35**, 408) in the following manner: The heating of the material with the respective reagents is performed always in the same Waltenberg's dish of 200 c.c. capacity. The liquids are drawn off by suction by means of an immersed inverted funnel covered with linen cloth or hardened filter-paper, the mouth of the funnel being covered with a finely perforated plate fused on to the body. Finally the treated cellulose is collected in a Gooch crucible, dried, weighed, incinerated, and weighed again. The method has been tested comparatively with those of Cross and Bevan and of König. Cross and Bevan's method gave higher results. The factors for comparing this method with Cross and Bevan's vary with different materials, and must be determined by future investigations. J. F. B.

Detection of Free Sulphuric Acid in Cotton. W. Zänker and K. Schnabel. (*Färber-Zeit.*, 1913, **24**, 260-263; through *Chem. Zentralbl.*, 1913, II., 907-908.)—Cotton absorbs sulphuric acid from dilute solutions, and retains traces of it so persistently that it is practically impossible to obtain a neutral product by simple washing with distilled water. Thus, in testing for minute traces of sulphuric acid in cotton, it is not sufficient to digest the fibre with water and test the extract. Such residual traces of acid are sufficient to bring about a marked weakening of the fibre when the latter is exposed to dry heat for any length of time. For the detection of small traces of acid the authors thoroughly moisten 2 to 3 grms. of the sample in a platinum dish, and then evaporate the water on the water-bath until the cotton retains about half its weight of moisture. A strip of violet litmus-paper is then wrapped up in the sample, and the whole is placed under pressure in a press. The quantity of moisture in the fibre should be sufficient just to damp the litmus-paper when the sample is left for some time under heavy pressure. In this way 0·25 mgrm. of sulphuric acid in 2·5 grms. of cotton, or 0·01 per cent., can be detected, whereas the aqueous extract from such cotton has no effect even on highly sensitive litmus-silk. With coloured samples the fibre should be moistened with alcohol and ether, and in this case the evaporation should be carried somewhat further than when water is employed; control tests must be carried out with pure cotton, in order to ascertain that the reagents themselves are free from acid. The destructive action of small traces of sulphuric acid on the cotton fibre only becomes evident after prolonged heating; under ordinary circumstances the strength of the material is even increased. If a sufficient quantity of the sample be available, 10 to 20 grms. may be digested with water in a platinum dish on the water-bath, and the liquid pressed out by means of a pestle. If this operation be performed repeatedly, the greater portion of the acid may be extracted even when the sample contains only 0·01 per cent. According to the authors, the quantity of free acid may be estimated by treating the sample with $\frac{N}{100}$ sodium hydroxide solution and titrating back the excess. In this manner 0·08 per cent. of sulphuric acid has been found in surgical cotton-wool, and as much as 0·17 per cent. in certain parchment-papers. J. F. B.

Estimation of Small Quantities of Wool in Cotton Goods. P. Heermann. (*Mitt. Kaiserl. Materialprüfungs-Amt Gross-Lichterfelde West*, 1913, **31**, 176, Abt. 3; through *Chem. Zentralbl.*, 1913, II., 999.)—The method depends upon the comparative insolubility of wool in sulphuric acid of strength suitable to dissolve the cotton. Five to ten grms. of the sample are boiled first with ether, and then with 96 per cent. alcohol, pressed, immediately immersed in from 20 to 100 times its weight (according to the quantity of sample taken) of cold sulphuric acid of 80 per cent. by weight, and well shaken, especially for the first hour. The cellulose is dissolved in three or four hours. The mixture is poured into an excess of water, and the insoluble portion collected on a fine copper sieve, and washed with water alone and with water containing ammonia. The air-dried wool is weighed, or it may be dried at 105° to 110° C., when 17 per cent., representing the normal moisture content, is added to its weight. O. E. M.

Estimation of Caoutchouc in Crude Rubber. R. Marquis and F. Heim. (*Bull. Soc. Chim.*, 1913, **13**, 862-866.)—The method depends on the action of sulphuric acid on caoutchouc in chloroform solution. One grm. of the rubber, freed from resins by previous treatment with acetone, is dissolved in 100 c.c. of chloroform, 8 grms. of pure sulphuric acid are added, the mixture is shaken for five minutes, and then poured into 200 c.c. of alcohol. The white flocculent precipitate which forms is collected on a filter, washed with alcohol until free from acid, dried at 100° C., and weighed. Its weight will be within 0·7 per cent. of that of the caoutchouc actually present. In a few cases the authors have applied the method to rubbers from which the resins have not been removed, the results obtained being satisfactory; but until this has been done with all the different varieties of crude rubber, it is advisable to remove the resins before the caoutchouc is estimated in any given sample.

W. P. S.

Volatile Constituents of Coal. Part III. A. H. Clark and R. V. Wheeler (*J. Chem. Soc.*, 1913, **103**, 1704-1713), **and with C. B. Platt** (*ibid.*, 1713-1715).—The authors have before expressed the view that coal contains two types of compounds of different degrees of ease of decomposition. The more easily decomposed type was designated "paraffin yielding," the less easily decomposed type "hydrogen yielding," as indicating the main gaseous products of their thermal decomposition. In the present paper experiments are described in which finely divided coal was fractionated by means of volatile solvents, and the separate fractions submitted to destructive distillation. The coal was first treated with pyridine in a Soxhlet extractor, and both the residue (80 per cent. of the whole) and the extract freed from pyridine were distilled at various temperatures. A portion of the pyridine extract was further fractionated by treatment with chloroform, which dissolved about a third of it, and the two fractions were freed from chloroform and distilled. The portion soluble in pyridine, but insoluble in chloroform, was intermediate in character between the portion insoluble in pyridine and the portion soluble in chloroform, but resembled the former much more closely in ultimate composition, proportion of volatile matter, and composition of gaseous products of destructive distillation. The portion soluble in chloroform is regarded as mainly derived from the resins and gums of the coal plants, the portion insoluble in pyridine as probably consisting of the degradation products of cellulose and the like. In addition to the distillation experiments with the coal fractions, similar ones were made with "dehydrated cellulose" prepared from filter-paper in the manner described by Cross and Bevan (*Phil. Mag.*, 1882, [v], **13**, 325). Distillation experiments were conducted in a platinum retort at 600°, 700°, 800°, 900°, and 1000° C. The following table gives the results at the extreme temperatures. The analyses of the materials subjected to distillation are calculated on the ash-free dry substance, as are the percentages of volatile and tarry matter and the volumes of gas obtained on distillation.

The portion of coal soluble in chloroform resembles ordinary colophony in that it powerfully affects a photographic plate (*cf.* Russell, *P. Roy. Soc.*, 1908, B, **80**, 376). When the powder was spread over an area as large as a threepenny piece on a photographic plate (film-side up) in the dark for sixteen hours, and the plate subsequently developed, not only did the small area come out black, but it was surrounded by a corona as large as a shilling. The unfractionated pyridine extract gave a sharp negative, but no corona, and the portion insoluble in pyridine had no visible effect on a photographic plate. Plane (not necessarily polished) surfaces of coal give negatives showing streaks and patches of different intensity. Apart from certain yellow markings, which are due to pyrites, these streaks and patches are attributed by the authors to those constituents of the coal which are resins or partly changed resins.

	Portion of Coal Insoluble in Pyridine.		Portion of Coal Soluble in Chloroform.		Dehydrated Cellulose.	
Proximate analysis :						
Volatile matter	35·0		77·3			
Fixed carbon ...	65·0		22·7			
Ultimate analysis :						
Carbon	80·8		85·3		61·3	
Hydrogen	5·2		7·1		3·7	
Oxygen	10·4		4·6		35·0	
Nitrogen	2·2		1·7			
Sulphur	1·4		1·3			
Distillation temperature °C.	600	1000	600	1000	600	1000
Total volatile matter ...	24·0	37·7	64·0	78·2	46·0	55·0
Tarry matter	7·0	3·8	56·0	42·9	2·0	2·0
C.c. gas (at N.T.P.) per gram	93·3	417·1	55·0	221·6	216·2	462·0
Gas analysis :						
Carbon dioxide ...	9·8	5·3	1·8	4·7	46·0	24·8
Ethylene	1·5	0·8	6·7	8·6	0·3	0·4
Carbon monoxide ...	14·3	30·8	4·7	10·6	30·2	41·3
Hydrogen	22·2	44·9	26·6	39·5	9·6	24·7
Methane	44·7	11·7	35·5	31·9	10·4	6·1
Ethane	5·5	2·3	18·6	3·5	1·2	0·6
Nitrogen	2·0	4·2	6·1	1·2	2·3	2·1

G. C. J.

Volatile Constituents of Coal. Part IV. Relative Inflammabilities of Coal Dusts. R. V. Wheeler. (*J. Chem. Soc.*, 1913, **103**, 1715-1722.)—It has been shown before that, of two coals, that which yields the higher percentage of "volatile matter" is not necessarily the more inflammable in the form of dust. If coal be regarded as a conglomerate of two main types of compounds, one yielding readily inflammable gases and vapours on heating to a comparatively low temperature, the other requiring a higher temperature of more prolonged duration to decompose it freely (see preceding abstract), the absence of any necessary connection between inflammability and total content of volatile matter might be expected. The direct determination of relative inflammability involves the use of elaborate apparatus of which a convenient type is described and illustrated in this paper. The author has previously suggested an indirect method based on the volume of paraffin hydrocarbons evolved by 1 gram of the ash-free dry coal when heated *in vacuo* at 650° C. It is now found that the percentage extractable by pyridine affords a very close measure of the inflammability of a coal. The coal is ground to pass a sieve of 94 meshes per lineal centimetre, and, after drying, from 3 to 10 grms. are extracted with pyridine for a week in a Soxhlet extractor. In the samples tried, all of which were bituminous coals with from 25 to 36 per cent. volatile matter, the pyridine extract ranged from 18 to 38 per cent., and a series of thirty experiments show a very close connection between pyridine extract and temperature of inflammation under standard conditions, a higher pyridine extract corresponding to a relatively highly inflammable coal. G. C. J.

Formic Acid as a Solvent. O. Aschan. (*Chem. Zeit.*, 1913, **37**, 1117-1118.) —Formic acid may be used as a solvent for many inorganic salts and organic compounds. For example, 100 grms. of the 95 per cent. acid will dissolve 16·5 grms. of sodium sulphate, 7·3 grms. of barium chloride, 75 grms. of barium iodide, and 43·1 grms. of calcium chloride at about 20° C. Certain aromatic hydrocarbon derivatives such as terephthalic acid, indigo, uric acid, and alizarine, are fairly soluble in cold formic acid, and are still more soluble in the boiling acid, so that these bodies may be crystallised from 95 per cent. formic acid. Uric acid, for example, forms microscopic rhombohedra, whilst alizarine forms large yellow needles. In the case of some compounds, such as suberic acid, citric acid, α-brom-camphor, α-nitronaphthalene, *m*-dinitrobenzene, etc., very large crystals are obtained. C. A. M.

Quantitative Separation of Nitrosubstitution Compounds from Nitroglycerine. A. L. Hyde. (*J. Amer. Chem. Soc.*, 1913, **35**, 1173.)—The method is a development of that already given (ANALYST, 1912, **37**, 572). When a mixture containing nitroglycerine and a nitro-compound is shaken with carbon disulphide and an acetic acid and water mixture, a partial separation is effected, the carbon disulphide layer containing a higher proportion of the nitro-compound than the original mixture, and the acetic acid layer a higher proportion of the nitroglycerine. The author's apparatus is designed to give a continuous fractional separation on this principle. It consists of thirteen upright tubes to hold the solvents, closed with double-bored rubber stoppers, and connected by tubes rising from the bottom of one tube and terminating in a fine jet below the stopper of the next. To the first tube and above it is attached a condenser and siphon measuring device; to the last, a siphon leading through a stopcock to a flask at a lower level, in which the solvent (the carbon disulphide) is evaporated, and rises in the form of vapour through a second tube to the condenser. The whole system is airtight. The first upright tube contains the sample dissolved in acetic acid and water; the next ten contain a layer of carbon disulphide and a layer of acetie acid and water; the last two contain water, to arrest any acetic acid carried over by the carbon disulphide. In operation a regulated stream of carbon disulphide from the condenser passes in fine drops through each tube in succession, carrying the nitro-compound, which collects in the evaporating flask with it. It also carries over some of the nitroglycerine, which is arrested by the acetic acid in succeeding tubes. Results of experiments show that separation of nitroglycerine from the nitrotoluenes, except crystallised trinitrotoluene, and from the nitrobenzenes and nitronaphthalenes, could be carried out satisfactorily. For trinitrotoluene a longer time, and refractionation of the mixture obtained, would be necessary. The method was also applied to the separation of nitrotoluenes from each other, and it is pointed out that it is applicable in general to other mixtures, provided that a pair of solvents of suitable properties can be found.

O. E. M.

Analysis of Black Powder and Dynamite. W. O. Snelling and C. G. Storm. (*Bulletin* 51, *Bureau of Mines*, U.S.A., 1913.)—This bulletin describes the methods of analysis applied by the Bureau of Mines to "ordinary" dynamite, the ammonia, gelatine, low-freezing, and granular dynamites, and the common grades of black gunpowder and blasting powder. "Permissible" explosives will be dealt with in a later bulletin. Physical examination, determination of gravimetric density (by displacement of sand in a stricken measure), tests for exudation and stability, sampling, with special reference to segregation of the nitroglycerine, chemical examination, and the determination of moisture and other constituents, are considered in detail for dynamite; the chemical analysis of the others, and the granulation and specific gravity of black powders, are briefly dealt with. O. E. M.

Composition of Arachis Oil. H. Meyer and R. Beer. (*Monatsh. für Chem.*, 1913, **34**, 1195-1208.)—Hehner and Mitchell (ANALYST, 1896, **21**, 328) isolated from arachis oil about 7 per cent. of a fatty acid which they considered from its melting-point to be stearic acid. The authors have repeated the estimation by means of a saturated solution of stearic acid, and have also obtained a fatty acid melting at 68° C. When, however, this was mixed with pure stearic acid of m.-pt. 69° C., the mixture melted at 62° to 63° C. By fractional crystallisation it was proved that the deposit from the saturated alcohol contained arachidic and lignoceric acids, but that no stearic acid was present. With regard to the unsaturated acids, no hypogæic acid could be detected in arachis oil, the only oxidation product yielded by the liquid fatty acids being α-sativic acid, derived from the oleic acid. C. A. M.

Candelilla Wax. H. Meyer and W. Soyka. (*Monatsh. für Chem.*, 1913, **34**, 1159-1172.)—The resinous constituent giving the Liebermann-Storch reaction was separated by extracting the wax with boiling 96 per cent. alcohol. On evaporating the alcoholic extract a yellow resinous mass was left, which, when distilled with zinc dust in a current of hydrogen, yielded a colourless sesquiterpene. The wax from which the resin had been removed consisted in the main of dotriacontane, $C_{32}H_{66}$ (m.-pt. 71° C.), and an oxylactone, $C_{30}H_{58}O_3$ (m.-pt. 88° C.), which closely resembled in its properties the oxylactone of lanoceric acid, isolated by Darmstaedter and

Lifschutz from wool fat. The sample of candelilla wax examined contained from 18 to 20 per cent. of resin (probably formed in the preparation of the wax), 74 to 76 per cent. of dotriacontane, and 5 to 6 per cent. of the oxylactone. C. A. M.

Estimation of Small Quantities of Mercury in Organic Substances. S. Lomholt and J. A. Christiansen. (*Comptes-rendus Trav. Lab.*, Carlsberg, 1913, **10**, 259-266.)—A method is described for the estimation of minute quantities (2 mgrms. or less) of mercury in such substances as urine, fæces, organs, etc., and consists in decomposing the organic matter, precipitating the mercury as sulphide after the addition of copper sulphate, dissolving the mixed sulphides, and depositing the mercury electrolytically on gold-foil. Solid substances are decomposed by heating with nitric acid; in case much fat is present, it is convenient to interrupt the oxidation, separate the layer of fat, and continue the oxidation of the liquid portion by the addition of permanganate. Urine is decomposed directly by heating it with the addition of permanganate. In either case the excess of permanganate introduced is destroyed by means of oxalic acid. About 0·05 grm. of crystallised copper sulphate is added to the clear solution, which is then partially neutralised with ammonia, and saturated with hydrogen sulphide. The mixed sulphides are collected on an asbestos filter, washed, and redissolved in a mixture of 5 c.c. of nitric acid and 1·5 c.c. of hydrochloric acid. The filter is washed, and the solution, which should measure about 40 c.c., is submitted to electrolysis. A platinum wire is used as the anode, whilst a piece of gold foil (1 × 2 cm.) soldered to a platinum wire serves as the cathode; a current of 1·5 volts and 1 milliampère is employed, and the electrolysis is continued for eighteen hours. The liquid is then drawn off, and the electrodes are rinsed by the addition of water while the current is still passing. After the gold cathode has been dried in a desiccator, it is weighed, then heated to redness in a tube through which a current of hydrogen is passed, and again weighed. The difference between the two weights gives the quantity of mercury present. The gold cathode must not be dried by washing it with alcohol and ether, as this treatment detaches an appreciable quantity of mercury, particularly when relatively large amounts are deposited. Traces of copper are also deposited with the mercury, and the volatilisation of this metal must be carried out in an atmosphere of hydrogen in order to prevent oxidation of the copper. W. P. S.

Estimation of Sulphur in Organic Substances. H. Apitsch. (*Zeitsch. angew. Chem.*, 1913, **26**, 503-504.)—The method consists essentially in burning the substances in a current of oxygen, absorbing the products of combustion in bromine, and estimating the resulting sulphuric acid. A weighed portion of the substance, contained in a platinum boat, is placed in a combustion tube, one end of which is connected with a supply of oxygen, whilst the other end is drawn out to form a narrow tube which passes through a rubber stopper into the absorption vessel. The latter consists of a Fresenius receiver containing sodium hydroxide solution to which has been added an excess of bromine; should this solution become colourless during the operation, more bromine must be added. Spirals of platinum gauze are placed in the combustion tube at a short distance on either side of the boat, and these spirals are maintained at a dull red heat during the combustion. The combustion tube must be free from any oily drops at the end of the combustion, but droplets of sulphuric acid may be ignored at this stage of the operation. After cooling, the tube, together with the boat and spirals, is washed out with water, the solution is added to the contents of the receiver, and the sulphuric acid is then estimated in the solution in the usual way. With suitable modification the method may be used for the estimation of sulphur in very volatile organic substances. W. P. S.

Quantitative Determination of Rosin in Paper. C. F. Sammet. (*J. Ind. and Eng. Chem.*, 1913, **5**, 732-735.)—Of the various methods investigated, the following gave the most reliable results: Five grms. of paper, cut into strips about ½ inch wide and folded into numerous tranverse folds, are placed in a Soxhlet extractor and covered with acidulated 83 per cent. alcohol, prepared by adding to 100 c.c. of 95 per cent. alcohol 15 c.c. of 5 per cent. aqueous acetic acid. The extraction flask is placed directly in the boiling water of a water-bath, and the rosin is extracted by distilling up the solvent from six to twelve times, according to the nature of the paper. The alcoholic extract, which may contain other bodies besides rosin, is transferred to a beaker, and evaporated on the water-bath to a bulk of a few c.c. The residue is cooled and taken up with about 25 c.c. of ether; the solution is transferred to a 300 c.c. separating funnel with about 150 c.c. of water containing a little sodium chloride to prevent emulsion; the liquid is shaken thoroughly and allowed to separate. The water is drawn off into a second funnel, and washed with another 25 c.c. of ether; the ethereal extracts are combined, and washed twice with 100 c.c. of water, or until the separation is sharp and the ether clear. If glue is present, it may be necessary to shake the ethereal extract with salt solution before washing with water. The washed ethereal extract is evaporated in a platinum dish, and dried at 98° to 100° C. for exactly one hour; prolonged heating causes a continual loss of rosin. It is found that, if acidulated 95 per cent. alcohol be used, the decomposition of the resinates is incomplete; an alcoholic strength of 83 to 86 per cent. gives the best results. Control tests have shown that the further quantity of rosin extracted by a second treatment is negligible for practical purposes, and the error involved by the process described above is within the limit of 0·20 per cent. Direct extraction of the paper with acidified ether tends generally to give low results, due to the slowness of extraction; but in certain cases the results are too high, owing to the separation of insoluble substances from the paper. J. F. B.

Use of Amalgamated Aluminium in the Estimation of Tannin. E. Kohn-Abrest. (*Ann. Chim. anal.*, 1913, **18**, 349-351.)—One hundred c.c. of the tannin solution are placed in contact with a piece of amalgamated aluminium-foil (*cf.* ANALYST, 1911, **36**, 516) about 1 mm. in thickness and weighing 3 grms. Contact is maintained for about five hours, or until all the tannin has been precipitated and the solution no longer gives a coloration with ferric chloride. If the solution is shaken continuously, the time required for the precipitation is only about one hour. The liquid is then filtered, the precipitate is washed, the filtrate diluted to a definite volume, and an aliquot portion is evaporated. The difference between the weight of this residue and that of the residue obtained on evaporating a corresponding volume of the original solution is equivalent to the amount of tannin precipitated. As a trace of aluminium is dissolved, a correction may be made for its amount by estimating the quantity of ash in the two residues. W. P. S.

INORGANIC ANALYSIS.

Precipitation of Aluminium Hydroxide, and Separation of Aluminium and Chromium. W. Jakób. (*Zeitsch. anal. Chem.*, 1913, **52**, 651-657.)—The precipitation of alumina from solutions of alkali aluminate by addition of bromine is complete only when the temperature is above 60° C., and the higher the temperature of precipitation, the more compact is the precipitate. If caustic alkali be added to a solution of aluminium nitrate until the precipitate at first formed redissolves, the solution heated to boiling, and excess of bromine-water added slowly so as not to reduce the temperature, the precipitate will not be gelatinous. If the mixture be boiled to expel bromine, filtered, and the precipitate well washed, the whole of the alumina, free from any trace of alkali, will be recovered. The best way to transfer to a filter particles of alumina which attach themselves to a beaker, is to treat the contents of the beaker with a little dilute nitric acid, which effects no visible change, but loosens the alumina from the glass. Ammonia is then added in excess, and the particles of alumina are washed on to the filter.

For the separation of aluminium from chromium, caustic alkali is added to the solution of the metals until the precipitate at first formed redissolves. Bromine-water is cautiously added to the cold solution until its green colour gives place to the yellow of a chromate solution. Chromium hydroxide may separate at first, but rapidly oxidises and redissolves. Finally the solution is heated to boiling, and more bromine-water added drop by drop to secure the precipitation of the alumina in a compact form. It always carries down some chromium hydroxide, from which, however, it can be completely freed as follows: The supernatant liquid is decanted through a filter, and the precipitate is boiled with water containing ammonia and ammonium nitrate, transferred to the filter, and washed there with water containing ammonia and ammonium nitrate. Test numbers obtained with pure solutions of aluminium nitrate and chromium chloride, and others obtained in presence of 1 or 2 per cent. of zinc and magnesium, are exact; but zinc and magnesium may come down with the alumina, and the method is not recommended in presence of these metals. Nor is it available in presence of sulphates or borates. G. C. J.

Differentiation of Natural and Artificial Barium Sulphate. J. Jámbor. (*Chem. Zeit.*, 1913, **37**, 1077.)—A method of distinguishing between natural and artificial barium sulphate is based upon the greater covering power of the latter. If equal quantities of the finely powdered sulphates are mixed with equal quantities of a dyestuff into a paste, the colour of the mixture prepared from the natural product will be of a lighter colour. The test may be made approximately quantitative by comparing the colour with those of standard mixtures containing up to 10 per cent. of the respective barium sulphates, the different pastes being pressed between glass plates with paraffin wax at the edges. Since differences of 2 per cent. and more may be observed with barium sulphates of different origin, the accuracy of the estimation is not greater than 5 per cent. It is noteworthy that a dyestuff mixture containing 1 per cent. of Water Blue and 0·5 per cent. of Auramine colours artificial barium sulphate violet, but the natural product green, and by varying the relative proportions of the two sulphates all shades between these colours may be obtained. In this way it is possible to distinguish variations of 1 per cent. C. A. M.

Characteristic Reaction of Bromine. I. Guareschi. (*Zeitsch. anal. Chem.*, 1913, **52**, 607-616.)—The author refers to his earlier papers (*Atti R. Accad. Sci. Torino*, 1912, **47**, 696, 988), and claims priority for the discovery that Schiff's aldehyde reagent affords a means of detecting minute quantities of bromine in presence of much chlorine and iodine (*cf.* Denigès, ANALYST, 1912, **37**, 581). The use of hydrogen peroxide, as suggested by Denigès, is unnecessary. The reagent is most conveniently employed distributed on starch-free paper; and if this is dried between other pieces of filter-paper, no free sulphur dioxide will remain, and less than 0·01 mgrm. of bromine is easily detected. For detecting traces of bromide in commercial chlorides and iodides, a considerable quantity of the substance is warmed with a 25 per cent. solution of chromic acid, and the vapours tested with filter-paper prepared as described. For detecting bromine in an organic compound of which it is a constituent, and not merely an accidental impurity, a fraction of a milligram suffices. The reagent may be made, not only from fuchsin, but from fuchsin S, rosaniline acetate, *p*-rosaniline hydrochloride, or Hofmann's violet, decolorised as described by Schiff, or preferably as described later by Gayon (see Denigès, *loc. cit.*). Hofmann's violet affords the most sensitive reagent, but the colour used must be the original colour of Hofmann—namely, trimethyl- or triethyl-pararosaniline chloride—and not methyl violet B extra, which is also sold as Hofmann's violet. The reaction depends on the formation of tetrabromo-derivatives of the dyestuff, and is readily distinguished from the reaction given by aldehydes. The latter reaction is given only in solution, and the reddish-violet colour is not removed from the solution by ether. When a liquid containing the tetrabromo-dyestuffs is shaken with ether, the violet-blue colour collects between the ethereal and aqueous layers.

G. C. J.

Volumetric Estimation of Calcium. T. Döring. (*Zeitsch. angew. Chem.*, 1913, **26**, 478-480.)—Dupré and Müller have thrown doubt on the accuracy of the method which depends on precipitation of the calcium as oxalate, and titration of the washed precipitate with permanganate (*Zeitsch. angew. Chem.*, 1902, **15**, 1244). According to them, the results may be 2 per cent. low. The author shows that quantities of calcium up to 0·1 grm. (the equivalent of 50 c.c. of $\frac{N}{10}$ permanganate) can be determined with an error not exceeding ±0·1 mgrm. Larger quantities should not be taken for analysis, as large precipitates are more difficult to wash, and calcium oxalate is soluble to the extent of 0·5 mgrm. in 100 c.c. of water, and water containing ammonium oxalate cannot be used for washing when the calcium is to be determined volumetrically. Large quantities may thus be under-estimated by

0·5 per cent. Indirect estimation as usually practised leads to results which are always high, usually more than 1 per cent. too high. This is due to the co-precipitation of ammonium oxalate with calcium oxalate. The indirect method gives fairly accurate results if the precipitate be thoroughly washed, but the necessity for such washing removes the sole advantage of the indirect method—namely, its greater rapidity. As it possesses the added disadvantage that two standard solutions are required, it cannot be recommended for exact work. G. C. J.

Cerium Dioxide as Contact Substance in Analysis by Combustion. J. Bekk. (*Ber.*, 1913, **46**, 2574-2579.)—Cerium dioxide distributed on asbestos is an effective substitute for the platinised asbestos of Dennstedt. Asbestos is steeped in a saturated solution of cerium nitrate, dried, and ignited. A 30-cm. length in the combustion-tube makes it possible to complete a combustion of 0·2 grm. of a volatile substance in generally less than twenty minutes from the time of beginning to heat the substance to the turning out of the flame. The catalyst does not retain halogens, which may be estimated simultaneously by placing in the forward part of the combustion-tube two tared boats containing reduced silver (Dennstedt), or, better, by means of a spiral of silver gauze wrapped round with platinum wire to guard against mechanical loss of silver halide during withdrawal. Nitrogen and sulphur are retained by lead peroxide placed in the forward part of the tube, as in Dennstedt's method; but sulphur cannot be determined simultaneously, as the catalyst retains an amount which varies with the temperature of combustion. The sulphur dioxide thus retained by the catalyst does not appreciably impair its efficiency, and can be expelled by prolonged and intense heating in a current of air. The catalyst is not poisoned by arsenic or by lead peroxide. After about fifty hours' effective use, the asbestos becomes reduced to a powdery condition. It is still usable, but the cost of its replacement is negligible. G. C. J.

Estimation of Copper by Means of Sodium Hypophosphite. J. Hanuš. (*Zeitsch. anal. Chem.*, 1913, **52**, 616-618.)—Dallimore (ANALYST, 1909, **34**, 452) described a modification of the Muthman-Mawrow method (*Zeitsch. anorg. Chem.*, 1896, **11**, 268) for the estimation of copper, and Windisch (ANALYST, 1913, 75) has recommended this modified method as exact. The author shows that it is open to the same criticism as the original Muthman-Mawrow method, which he and Soukup found (ANALYST, 1911, **36**, 302) to be dependent on approximate compensation of errors, which are individually large, and which may be very far from being mutually compensating. That Windisch did not find copper in all his filtrates—he found it in some—is probably due to his testing the first filtrate, and not the subsequent wash-waters, which always contain much more copper than the first filtrate. Better results are obtained by neutralising exactly with $\frac{N}{1}$ sodium hydroxide, using phenolphthaleïn as indicator, as soon as precipitation is complete, followed by rapid filtration and washing. Even then the method is useless for the exact determination of copper in presence of cadmium and zinc. As a technical method for the valuation of commercial copper sulphate, using large quantities as recommended by Dallimore, it may have some value. G. C. J.

Estimation of Copper by Means of Sodium Hypophosphite. R. Windisch. (*Zeitsch. anal. Chem.*, 1913, **52**, 619-628.)—The author seeks to show that the method described by Dallimore, and recommended by him, is free from the objections raised by Hanuš and Soukup to the original Muthman-Mawrow method (*cf.* preceding abstract). Pure solutions of copper sulphate were precipitated in the manner previously described, and the ignited precipitates proved to be substantially pure copper oxide. The filtrates were proved to contain so little copper that the error due to incomplete precipitation is estimated as less than 0·2 per cent., but the wash-waters do not appear to have been examined (*cf.* preceding abstract). A sample of Merck's copper sulphate was found to contain 99·16 per cent. of $CuSO_4·5H_2O$ as the mean of sixty-four experiments, but the individual results range from 98·76 to 99·55.

G. C. J.

Determination of Oxygen in Copper and Brass. T. West. (*J. Inst. Metals*, advance proof issued August, 1913.)—Turner has shown (*J. Inst. Metals*, 1912, **8**, 248) that any oxygen in brass probably exists as zinc oxide, and therefore cannot be determined by the methods adopted for determining oxygen in copper; for, though the zinc oxide is reduced by hydrogen at a high temperature, in the cooler portion of the combustion-tube the reverse reaction occurs. The author has had resort to reduction in a current of carbon monoxide. The method was first applied to copper, and, after results had been obtained which agreed closely with those obtained by reduction in hydrogen, further experiments were made with zinc oxide. When the conditions for the accurate estimation of oxygen in zinc oxide had been determined, some brasses were examined, with the result that it is shown that brass of good quality contains on an average 0·002 to 0·003 per cent. of oxygen.

The combustion-tube used was of clear silica, and rested in the porcelain tube of an electric furnace. The carbon monoxide was generated from sodium formate and sulphuric acid, and stored in a gas-holder, between which and the combustion-tube were (1) a caustic potash wash-bottle, (2) a tube containing solid potash, (3) a heated Jena glass tube containing copper gauze, (4) another potash wash-bottle, (5) a sulphuric acid wash-bottle, (6) another heated glass tube containing copper, (7) another tube containing solid potash, and (8) another sulphuric acid wash-bottle. The carbon dioxide resulting from the combustion was absorbed by potash contained in Geissler bulbs provided with a small calcium chloride tube, and the train of apparatus was terminated by a guard-tube containing sulphuric acid. The temperature was controlled by a rheostat and measured by a thermocouple, the wires of which passed between the porcelain and silica tubes. In use, the boat containing the drillings is introduced into the combustion-tube, and a current of carbon monoxide is passed through the apparatus until the Geissler bulbs show no change in weight in one hour. The temperature is then raised to 1,050° C., and maintained there for one and a half hours, after which the electric circuit is broken, the apparatus allowed to cool for thirty minutes without checking the stream of gas, and the Geissler bulbs are weighed full of carbon monoxide.

Experiments with zinc oxide showed that the use of too slow a gas current might lead to results much below the truth, but with three bubbles per second exact results were obtained.

The method is not available for copper-zinc alloys containing tin or nickel, as such alloys decompose carbon monoxide, although tin alone does not do so.

G. C. J.

Volumetric Estimation of Fluorine. A. Greef. (*Ber.*, 1913, **46**, 2511-2513.) —The method depends on the fact that, when sufficient ferric chloride is added to a neutral solution of an alkali fluoride, the whole of the fluorine separates as a white insoluble fluoride of the type Na_3FeF_6. The end-point of a simple titration with thiocyanate as indicator is not easily seen, as no intense red, but only a particular shade of yellow, marks it. The use of ether and alcohol gives the characteristic red colour in the ethereal layer, but the end-point is only sharp when the slight solubility of the precipitate is still further repressed by large addition of sodium chloride.

To 25 c.c. of a neutral solution of an alkali fluoride, 20 grms. of sodium chloride and 5 c.c. of a 20 per cent. solution of potassium thiocyanate are added. The mixture is then titrated with ferric chloride solution (1 c.c. = 0·01 grm. NaF) until a faint yellow colour appears. Alcohol (10 c.c.) and ether (10 c.c.) are then added, and the titration continued, with vigorous agitation after each addition, until the ethereal layer acquires a persistent red colour.

Commercial sodium fluoride usually has an acid reaction, and contains sodium hydrogen fluoride and sodium silicofluoride. On titration with sodium hydroxide, using phenolphthaleïn as indicator, these compounds react as follows:

$$NaF{\cdot}HF + NaOH = 2NaF + H_2O \text{ and } Na_2SiF_6 + 4NaOH = 6NaF + SiO_2 + 2H_2O.$$

As the neutralised solution contains the whole of the fluorine as sodium fluoride, total fluorine may be estimated in the manner above described. The sodium hydrogen fluoride is determined in a separate portion of the sample as follows: The substance (0·5 grm.) is dissolved together with an equal weight of potassium chloride in 25 c.c. hot water, the solution cooled, 20 c.c. alcohol added to render potassium silicofluoride insoluble, and the sodium hydrogen fluoride titrated with $\frac{N}{10}$ sodium hydroxide, using phenolphthaleïn as indicator. The percentage of sodium hydrogen fluoride being known, the percentage of sodium silicofluoride can be calculated from the amount of alkali required to neutralise the solution taken for the estimation of total fluorine.

The following are typical results: Present 0·6 grm. NaF, 0·2 grm. NaF·HF, 0·2 grm. Na_2SiF_6; found 0·598 grm. NaF, 0·201 grm. NaF·HF, 0·201 grm. Na_2SiF_6.

G. C. J.

Method of Analysing Hypochlorites. F. Ducelliez. (*Ann. Chim. anal.*, 1913, **18**, 345-349.)—When cobalt chloride is brought into contact with calcium hypochlorite, oxygen is evolved and cobalt peroxide is precipitated; under the conditions given below, this reaction may be applied to the estimation of hypochlorites. Two grms. of the calcium hypochlorite under examination are placed in a test-tube together with 50 c.c. of boiled water; the test-tube is closed with a rubber stopper carrying a delivery-tube, which is connected by a length of rubber tubing with a measuring burette supported in a vessel of water. The test-tube is first held in an inclined position while a quantity of 0·05 grm. of crystallised cobalt chloride is placed in it just below its mouth; the stopper is then inserted, the cobalt chloride is caused to fall into the solution, and the test-tube is immersed in a boiling water-bath, the evolved oxygen being collected in the burette. At the end of about thirty minutes the test-tube is cooled, placed in the water in the vessel containing the burette, and the volume of oxygen is read off. The volume of gas found is multiplied by 0·93 to correct for vapour pressure of water and temperature, provided that the temperature of the water is 15° C., and to the result is added 1·2 c.c., this being the volume of oxygen absorbed by the cobalt oxide to produce cobalt peroxide. The method may be applied to alkali hypochlorites, and the results obtained agree with those found by the iodometric process. W. P. S.

New Method for the Determination of the Concentration of Hydroxyl Ions. F. Francis and F. H. Geake. (*J. Chem. Soc.*, 1913, **103**, 1722-1734.)—The rate of decomposition of nitrosotriacetonamine into nitrogen and phorone under the action of alkalis affords a measure of the concentration of hydroxyl ions. The velocity constants have been established in presence of aqueous solutions of sodium, potassium, and barium hydroxides, and found to be proportional, not to the concentrations of the bases themselves, but to the concentration of hydroxyl ions. In any individual experiment the values of the velocity constant agree to within 1 to 2 per cent., and the results are reproducible to within 2 to 3 per cent. The method is available with this degree of accuracy for the estimation of the concentration of hydroxyl ions up to 0·05 *N*, and the general formula may be expressed as follows: Concentration of $OH' = K_{t^\circ}/R$, where $R = 1{\cdot}96 \times 2{\cdot}20^{\frac{t^\circ - 30}{10}}$; K_{t° is the observed constant at the temperature t°. Between concentrations of 0·05 *N* and 0·3 *N* hydroxyl ion, the unimolecular constants "drift," and the method is inapplicable within these limits. The drift ceases at the latter concentration; from that point up to a concentration of 1·4 *N* the accuracy is diminished, and beyond that considerably diminished, since the velocity constants change only slightly with large increases in the concentration of the hydroxyl ions near the higher limits. The effect of the presence of neutral salts in moderate quantities is unimportant. J. F. B.

Valuation of Hydrated Oxide of Iron by Means of the "Sulphuretted Hydrogen Burette." E. Linder. (*Forty-ninth Annual Report on Alkali, etc., Works, Proceedings during* 1912, pub. 1913, 20-26.)—The method consists in treating a small weighed quantity of air-dried and moistened oxide (0·6 to 1 grm. oxide containing 15 to 20 per cent. moisture) with excess of sulphuretted hydrogen. The oxide is contained in a small glass cup floating on the surface of mercury in a "laboratory vessel" (the bell-shaped piece of apparatus so called by designers of apparatus for exact gas analysis). The sulphuretted hydrogen is measured in a burette over mercury. To eliminate air from the upper part of the laboratory vessel, the cup is made as small as possible, and its neck constricted to follow the curvature of the upper part of the laboratory vessel, into which it can in consequence rise. The body of air is further reduced by causing the mercury to rise in the annulus between the cup and the walls of the laboratory vessel as far as a mark on the neck of the latter. By this means the working space is reduced to about 15 c.c., and the air in this space is displaced by hydrogen before the sulphuretted hydrogen is admitted. By thus working with a constant volume of an inert gas in the laboratory vessel, the contraction that ensues during the sulphiding of the oxide is made an exact measure of the weight of sulphur absorbed, as temperature and pressure corrections for 15 c.c. of gas are insignificant in comparison with the large volume (150 to 200 c.c.) of sulphuretted hydrogen absorbed by the oxide, and can easily be calculated and allowed for if desired.

The sulphuretted hydrogen burette is water-jacketed, and is partly of wide, partly of narrow, bore, so that any quantity of gas from 140 to 250 c.c. can be measured accurately to within 0·1 c.c. The rest of the apparatus includes levelling slide, rising table, and generators for hydrogen and hydrogen sulphide, and is fully illustrated in the original, which also describes the method of operation, including the determination of the gas space above the mercury in the laboratory vessel, determination of a correction factor for the solubility of hydrogen sulphide in the water originally present and that produced during the sulphiding operation, and analysis of the residual gas when this is necessary, as with oxides containing carbonates.

The merits of this method are best considered by reference to a standard method, such as that described in Lunge and Keane's "Technical Methods of Chemical Analysis," vol. ii., part ii., p. 772. The advantage of the method there described lies in the fact that a large weight of material can be operated on. But air is not effectually excluded, and no account is taken of carbon dioxide, which is freely liberated from some oxides and lost in the residual gases. Moreover, that method is not well suited for determining the maximum efficiency of oxides which absorb sulphuretted hydrogen over a period of six or seven days. In purifiers which remain undisturbed for many days, the bottom layers of oxide are in contact with gas for prolonged periods, and a measure of the maximum efficiency attained on exhaustive exposure in a desideratum. A gravimetric method with apparatus involving many parts, and necessitating disconnection for weighing from time to time, is less convenient than the volumetric method now described, and, finally, the use of dry air at the end of an experiment to displace residual gases is inadmissible in exact analysis.

G. C. J.

Detection of Molybdenum. E. Kedesdy. (*Mitt. Kaiserl. Materialprüfungs-Amt, Gross-Lichterfelde West,* 1913, **31**, 173; through *Chem. Zentralbl.,* 1913, II., 996.)—The detection of molybdenum by means of potassium thiocyanate is recommended by Treadwell (*Kurzes Lehrbuch der analyt. Chem.*), who states that there is no reaction with acid molybdate solutions, but on addition of zinc or stannous chloride a blood-red coloration of molybdenum thiocyanate is produced, even if phosphoric acid is present, which distinguishes molybdenum from iron. The molybdenum thiocyanate is taken up by ether on shaking. As hexavalent molybdenum does not give the reaction, it is better to effect reduction before carrying out the reaction, and also when the valency of the molybdenum is unknown. The reduction is also necessary in order to convert any ferric compounds to ferrous, since phosphoric acid or phosphate not only checks the reaction of thiocyanate with iron, but also with molybdenum. If the reduction is carried too far, the reaction may fail altogether or be much weakened. Commercial ether usually contains peroxide, which would cause the reoxidation of small quantities of ferrous to ferric iron. In carrying out the reaction, iron should be as far as possible excluded, over-reduction of the molybdenum avoided, and ether freshly distilled from stannous chloride alone used. O. E. M.

Action of an Excess of a Soluble Ferrocyanide on Solutions of Zinc, Copper, and Nickel, and a Volumetric Method for the Estimation of Nickel. R. Meurice. (*Ann. Chim. anal.*, 1913, **18**, 342-345.)—Zinc salts, when treated with an excess of potassium ferrocyanide, yield a definite compound having the composition $Zn_3K_2[Fe(CN)_6]_2$, and the reaction may be employed for the volumetric estimation of this metal. The zinc solution is treated with an excess of potassium ferrocyanide solution, diluted to a definite volume, the precipitate is allowed to settle, and an aliquot portion of the clear supernatant liquid is titrated with potassium permanganate solution after the addition of sulphuric acid. The ferrocyanide solution is standardised against the permanganate solution, and this in turn against pure iron. In the case of copper, the ferrocyanide precipitate varies in composition with the concentration of the solutions, and is, moreover, slightly soluble, so the above method cannot be applied to the estimation of copper. Nickel, however, yields a precipitate having the composition $NiK_2Fe(CN)_6$, but only under certain conditions—namely, in the presence of an excess of ferrocyanide and of ammonium sulphate. For the estimation of nickel, the solution, containing about 0·1 grm. of the metal, is treated with 100 c.c. of a 3·5 per cent. potassium ferrocyanide solution, 5 grms. of ammonium sulphate are added, the mixture is diluted to 200 c.c., filtered, and an aliquot portion of the filtrate is diluted with seven times its volume of water, acidified with sulphuric acid, and titrated with permanganate solution. One atom of iron is equivalent to 1 atom of nickel. W. P. S.

Separation of Palladium from Gold, Platinum, Rhodium, and Iridium. M. Wunder and V. Thüringer. (*Zeitsch. anal. Chem.*, 1913, **52**, 660-664.)—Palladium and gold can be separated from the other metals of the platinum group, except platinum itself, by precipitation from weak (1 per cent.) hydrochloric acid solution by means of dimethylglyoxime. The reagent is dissolved in boiling water and added to the boiling solution, which is maintained in ebullition for some time, and then allowed to cool. The precipitate is collected on a hardened filter and washed with 1 per cent. hydrochloric acid. The filter and its contents are then cautiously incinerated (*cf.* ANALYST, 1913, 79) and the residue finally heated strongly with a blowpipe flame. The metal sponge is dissolved in a few drops of *aqua regia*, and the solution repeatedly evaporated to dryness with hydrochloric acid. The residue is dissolved in water, 1 to 2 grms. ammonium oxalate are added, and the mixture digested at 60° C. for some hours. Dilute sulphuric acid (10 to 15 c.c.) is added to assist filtration, and the gold is filtered off on a hardened filter, washed first with water containing sulphuric acid, then with 1 per cent. hydrochloric acid, dried and ignited. The filtrate is nearly neutralised with ammonia and the palladium reprecipitated by means of dimethylglyoxime. After cautious incineration of the filter-paper, the palladium is reduced in hydrogen and allowed to cool in a current of carbon dioxide.

Platinum must be separated as ammonium platinic-chloride before palladium is precipitated by means of dimethylglyoxime. The neutral solution is concentrated to small bulk, saturated with ammonium chloride, and left for two days. The precipitate is washed with a saturated solution of ammonium chloride, dried, the filter cautiously incinerated, the residue strongly ignited at first with access of air, then in a current of hydrogen, and finally cooled in a current of carbon dioxide. The palladium is determined in the filtrate in the manner already described.

Palladium is separated from rhodium by precipitation with dimethylglyoxime. The rhodium in the filtrate is determined as follows: The filtrate is concentrated to small bulk, hydrochloric acid and a pinch of sodium chlorate are added, and the mixture heated until chlorine is expelled. This destroys the dimethylglyoxime, which might interfere with the subsequent reduction of the rhodium. The solution is diluted and the rhodium reduced by repeated addition of small pieces of magnesium. When the solution is colourless and all magnesium apparently dissolved, the rhodium is filtered off, washed with 5 per cent. sulphuric acid, to remove traces of magnesium, ignited, reduced in hydrogen, and cooled in a current of carbon dioxide. The separation of palladium from iridium and subsequent determination of the iridium is carried out in an exactly similar manner, the destruction of the dimethylglyoxime in this case being absolutely necessary.
G. C. J.

Quantitative Volatilisation and Separation of Phosphoric Acid from the Metals of the Copper Group, and from Aluminium, Tin, and the Alkali Metals.

P. Jannasch and R. Leiste. (*J. f. prakt. Chem.*, 1913, **88**, 129; through *Chem. Zentralbl.*, 1913, II., 994.)—In continuation of previous work (ANALYST, 1909, **34**, 507) it was found that the volatilisation of the phosphoric acid may be represented thus:

$$1.\ P_2O_5 + 2CCl_4 = 2POCl_3 + COCl_2 + CO_2.$$
$$2.\ 2P_2O_5 + 3CCl_4 = 4POCl_3 + 3CO_2.$$
$$3.\ MePO_4 + 2CCl_4 = MeCl_3 + POCl_3 + COCl_2 + CO_2.$$

While the secondary splitting up of the carbon tetrachloride takes place thus:

$$4.\ 2CCl_4 = C_2Cl_4 + 2Cl_2,$$
and
$$5.\ 2CCl_4 = C_2Cl_6 + Cl_2.$$

In all reactions at a high temperature free chlorine and hexachloroethane were formed, but not at temperatures below a red heat. The quantitative decomposition of a phosphate, thus due to carbon tetrachloride alone, is hindered by the presence of chlorine, to remove which a new method, involving the use of intermittent currents—was employed; this also prevents the regeneration of phosphoric acid in the distillation-tube. The following conditions were found necessary for the complete separation of phosphoric acid from the bases: (1) The use of an asbestos plug to hold back the metallic chloride; (2) the use of a diluting agent, such as quartz powder, to prevent the substance from fusing together; (3) complete absence of moisture. The methods and apparatus used for the separation of phosphoric acid from copper, silver, lead, cadmium, mercury, bismuth, aluminium, tin, and the alkali metals, by means of the stream of carbon tetrachloride, are described in detail. The addition of quartz powder renders the method applicable to phosphates of the alkaline earth and alkali metals, and the ammonium sulphide and hydrogen sulphide groups. Aluminium and tin, however, require the addition of potassium chloride also. O. E. M.

Estimation of Phosphoric Acid Soluble in Citric Acid in Thomas Slag. M. Popp. (*Chem. Zeit.*, 1913, **37**, 1085-1087.)—It has been proposed previously (*cf.* ANALYST, 1913, 171), in the estimation of phosphoric acid in Thomas slag, to prevent the interfering action of dissolved silica by the addition of ferric chloride, hydrogen peroxide being also added in order to oxidise any hydrogen sulphide which may be present, and the author now gives details of a method embodying these principles. Fifty c.c. of the citric acid extract of the slag are treated with 25 c.c. of iron-ammonium citrate solution and 1 c.c. of 3 per cent. hydrogen peroxide; more of the latter must be added if the mixture is still dark in colour owing to the presence of unoxidised ferrous sulphide. Twenty-five c.c. of magnesia mixture are then added, and the estimation is continued in the usual way. The iron-ammonium citrate solution is prepared by mixing 1 kilo of citric acid with 30 grms. of ferric chloride dissolved in 50 c.c. of water, adding 4 litres of 20 per cent. ammonia, and, when solution is effected, diluting the whole with water to 5 litres. The results are usually within 0·3 per cent. of those found when the silica has been removed by means of hydrochloric acid. W. P. S.

Volumetric Estimation of Phosphorus in Steel by Macagno's Method. H. Wdowiszewski. (*Chem. Zeit.*, 1913, **37**, 1069-1071.)—Macagno (*Gazzetta*, 1874, **4**, 467) proposed to estimate phosphorus by reducing the phosphomolybdate precipitate by means of zinc in sulphuric acid solution, and titrating the resulting molybdenum sesquioxide with permanganate. As modified by Emmerton (*Zeitsch. anal. Chem.*, 1892, **31**, 71), who concluded that the final product of reduction by zinc was $Mo_{12}O_{19}$, and not Mo_2O_3, the method is given in many textbooks—for example, in Blair's "Chemical Analysis of Iron." The method, as described by Blair, gives rise to results which may be 100 per cent. too high, but are more commonly from 10 to 30 per cent. too low. The author finds that it is essential to success to wash the phosphomolybdate precipitate with water after washing it with dilute nitric acid, as otherwise ammonium nitrate will pass into solution when the precipitate is dissolved in ammonia, and this nitrate will be reduced by the zinc to nitrite, which may double the consumption of permanganate in the final titration. The time allowed by Blair (ten minutes) and others for reduction is much too short, but molybdic acid can be reduced to Mo_2O_3 by zinc. On the other hand, the reduced solution is so rapidly oxidised beyond the stage of the hypothetical $Mo_{12}O_{19}$ that accurate results cannot be obtained by any method involving filtration from undissolved zinc or direct titration with permanganate. By decanting from the zinc into a measured and more than sufficient volume of standard permanganate and titrating back with oxalic acid, excellent results can be obtained.

The phosphomolybdate precipitate, obtained in any manner which ensures its having a $P_2O_5 : MoO_3$ ratio closely approximating 1:24, is freed from iron by washing with 1 per cent. nitric acid, and from nitric acid by washing with water. The filter is set in the neck of the 300 c.c. conical flask used for precipitation, the precipitate dissolved in a little 4 per cent. ammonia, and the filter washed with hot water until the contents of the flask amount to about 30 c.c. From 80 to 100 c.c. dilute (1:4) sulphuric acid and 10 grms. zinc are added, and the mixture is maintained just short of ebullition for forty-five to fifty minutes, which is only about fifteen minutes longer than is absolutely necessary. It is then removed from the hot-plate, and the clear liquid immediately decanted into another flask containing a measured volume of standard permanganate. The flask and zinc are rinsed with hot 10 per cent. sulphuric acid, which is also decanted into the flask containing the permanganate, and the excess of permanganate is titrated with an oxalic solution of equivalent strength.

The test numbers agree well with those obtained on large quantities of steel by the use with every precaution of the gravimetric (magnesia) method, and still more closely with results obtained on the same steels by the alkalimetric method. A single determination cannot be completed in much less than two hours from weighing the drillings, but several can be conducted simultaneously. G. C. J.

Estimation of Cobalt and Uranium in Steel. H. König. (*Chem. Zeit.*, 1913, **37**, 1106-1107.)—The method for the determination of cobalt depends on the electrode position of cobalt, nickel, and iron from an ammonium oxalate electrolyte, the separate estimation of iron and nickel, and calculation of cobalt by difference. The metallic deposit after being weighed is used for the determination of iron as follows: It is dissolved in hot dilute sulphuric acid, and nickel sulphate solution is added cautiously until its green colour just predominates over the pink due to cobalt. The iron can then be titrated with permanganate as usual. Contrary to the statements of Brunck (*Stahl. u. Eisen*, 1908, 331) and others, nickel cannot be separated from cobalt by means of dimethylglyoxime. In steels containing nickel and cobalt, the former can be determined as follows: Tungstic acid is separated as usual by means of *aqua regia*, most of the iron removed from the solution by shaking with ether, dissolved ether is boiled away, the solution reduced by means of sulphurous acid, the excess of the latter boiled away, and the solution neutralised with potassium hydroxide. When cool, potassium cyanide solution is added, whereby nickel, cobalt, and iron go into solution, but chromium is precipitated. The mixture is made up to 200 c.c., mixed, filtered, and 100 c.c. taken for the estimation of nickel. This 100 c.c. is boiled, whereby the potassium cobaltocyanide is transformed for the most part into cobalticyanide, which is not precipitated by potassium hydroxide and bromine water. Addition of bromine water to the cooled solution completes this transformation and precipitates the nickel as hydroxide. The nickel hydroxide is filtered off, washed, dissolved in dilute hydrochloric acid, chlorine expelled by boiling, and the nickel precipitated by means of dimethylglyoxime.

For the concentration of small quantities of uranium from solutions containing much iron, advantage is taken of the fact that uranium is not deposited from an ammonium oxalate electrolyte. The steel is treated with *aqua regia* to separate tungsten, chlorides are decomposed by evaporation with sulphuric acid, and the iron is deposited electrolytically after neutralisation of excess of acid by ammonia and addition of ammonium oxalate. After the iron has been deposited, the electrolysis is continued to destroy ammonium oxalate. The solution usually acquires a yellow colour due to formation of chromate, and a precipitate containing uranium as well as chromium, manganese, and vanadium, and a trace of iron may separate. The solution is boiled down to one-third of its bulk to destroy ammonium carbonate, which results from the decomposition of the oxalate, and the precipitate containing all the uranium together with more or less chromium, aluminium, manganese, iron, and vanadium, is filtered off and washed with a dilute solution of ammonium chloride. It is then dissolved in dilute hydrochloric acid, any chromic acid present is reduced by addition of a few drops of alcohol, and the solution boiled with a little nitric acid and transferred to a small flask in which it is cooled. To the cold liquid, excess of ammonium carbonate and ammonium sulphide are added, and the flask is corked and set aside for twelve hours. Iron, chromium, manganese, and aluminium are thus precipitated, and in the filtrate uranium is separated from vanadium by acidifying with acetic acid and heating to boiling, when vanadium comes down as sulphide. The filtrate from the vanadium is reoxidised by boiling with nitric acid, uranium is precipitated by means of ammonia, the precipitate washed with dilute ammonium chloride, and the uranium finally weighed as U_3O_8, or as UO_2. The author prefers to ignite the precipitate in a Rose crucible in a current of hydrogen and weigh as UO_2. G. C. J.

Preparing Sections of Fractures of Steel for Microscopic Examination. A. Campion and J. M. Ferguson. (*J. Iron and Steel Inst.*, 1913, Advance Proof.) —In investigations relating to the breakdown of metals, an examination of the fracture or of a section through the fracture is often of importance. The preparation of sections of fractures for microscopical examination, especially of soft and ductile materials, presents considerable difficulty, as during the polishing operations the edges of the fracture become rounded or worn away. In order to overcome this difficulty, Rosenhain embedded the fracture in copper by electro-deposition and then cut through the copper and steel in the desired direction. The method is excellent, but requires much attention and considerable time for the deposition of $\frac{1}{8}$ inch of copper. The authors find that satisfactory results may be obtained by embedding the fractured end of a test bar in a suitable fusible alloy, cutting a section and polishing. Of several alloys tried, the following was found the best for general work: Bismuth 50, lead 30, tin 25, zinc 3; m.-pt. below 100° C. This alloy readily penetrates the minute fissures in the fracture, is hard enough to support the edges of the fracture during polishing, and adheres strongly to the specimen. The fractured test-piece is dipped momentarily into dilute (1 : 1) hydrochloric acid, then into similar acid which has been "killed" by zinc, and finally into the alloy a little above its melting-point. If the alloy freezes, the test-piece is held in position until it melts again. It is then allowed to cool, still holding the test-piece until solidification takes place. When cold, sections are cut by means of a fine saw and the specimen polished in the usual manner. A section can be prepared and a photograph obtained within half an hour of the breaking test. G. C. J.

Rapid Estimation of Sulphur in Burned Pyrites. L. Sznajder. (*Chem. Zeit.*, 1913, **37**, 1107.)—2·5 grms. of the finely divided material is mixed with about 1 grm. sodium carbonate and 4 grms. zinc oxide in a spun iron crucible, and kept at a red heat for thirty minutes. The cooled contents of the crucible are transferred to a 250 c.c. flask with hot water, the flask is filled to the mark, and its contents are shaken and filtered. Two hundred c.c. of the filtrate is heated to boiling, and exactly neutralised to phenolphthaleïn by addition of dilute hydrochloric acid to the boiling solution. Thirty c.c. of $\frac{N}{5}$ barium chloride solution are added, whilst maintaining violent agitation of the liquid, and the excess of barium chloride is then titrated with $\frac{N}{5}$ sodium carbonate. In spite of the presence of the precipitate, the first appearance of a pink tinge is readily recognised after a little practice. The method gives results about 1 per cent. higher than those obtained gravimetrically, but on material with only 2 to 3 per cent. of sulphur this is negligible. G. C. J.

Quantitative Precipitation of Tungstic Acid by Means of Aromatic Amines. E. Kafka. (*Zeitsch. anal. Chem.*, 1913, **52**, 601-606.)—It is known that tungstic acid can be precipitated by means of benzidine and α-naphthylamine (v. Knorre, *Ber.*, 1905, **38**, 783; Tschilikin, *Ber.*, 1909, **42**, 1302). The author now shows that cumidine, tetramethylparadiaminodiphenylmethane, and tetramethyldiaminobenzophenone can be used for the purpose. The reagents are prepared as follows: Cumidine (5 grms.) is mixed with 10 c.c. water and dissolved by addition of 5 c.c. concentrated hydrochloric acid, or 6 grms. tetramethyldiaminodiphenylmethane is dissolved in 4 c.c. hydrochloric acid, or 7·5 grms. recrystallised tetramethyldiaminobenzophenone is dissolved in 10 c.c. hydrochloric acid, and the solutions are diluted to 100 c.c. As the precipitates are somewhat soluble in water, but much less so in dilute solutions of the reagents, they are washed with a solution made by diluting 5 c.c. of the reagent to 100 c.c. To 50 c.c. of a solution containing about 0·2 grm. tungstic acid as sodium tungstate, 10 to 15 c.c. of the reagent is added, with constant stirring. The mixture is stirred from time to time during an hour, at the end of which time the precipitate is filtered off, washed, ignited in a platinum crucible without previous drying, and weighed as WO_3. Test numbers obtained with solutions of pure sodium tungstate are accurate within ±0·8 per cent. The precipitates cannot be dried without partial decomposition, but analysis of such dried precipitates leaves no doubt that the original precipitate in the case of tetramethyldiaminodiphenylmethane has the composition $WO_3.C_{17}H_{22}N_2$, and in the case of tetramethyldiaminobenzophenone $2WO_3.3C_{17}H_{20}N_2O$. G. C. J.

Action of Various Waters on Lead. H. Heap. (*J. Soc. Chem. Ind.*, 1913, **32**, 771-775, 811-815, 847-856.)—Experiments were made by a modification of Frankland's lead-pipe method and by a method depending on the use of pure lead foil. At an early stage in the research it was found that lead pipes could not be used continuously for a series of comparable experiments with different waters. Once they had acquired a protective coating from one water, they resisted the action of other water which would have attacked clean lead: the protective coating gradually dissolves in a corrosive water, with the result that corrosion is slow at first and then accelerates. The disadvantages of using new lead pipe for each experiment are obvious, and fortunately it was found that such experiments gave results in close agreement with those made by the foil method, which was consequently adopted in all the later work.

The foil (0·012 cm. thick) was polished brightly with wash-leather and cut into strips 6·35 by 2·54 cms. In each experiment one of these strips was placed in a specially constructed glass vessel entirely filled with the water to be tested. The vessels were cylindrical in shape, held about 120 c.c., and were fitted with broad conical stoppers, so that by filling the vessel with water and inserting the stopper, water was displaced until the stopper fitted tightly. In experiments made at temperatures far removed from room-temperature, the water in the bottle was allowed to reach the temperature of the thermostat and the foil and stopper then inserted. In some experiments the water was placed under anaerobic conditions. The gases were pumped off by making use of Adeney's gas analysis apparatus, which offers the further advantage that various gases can be introduced at definite pressures to the gas-free water.

The most important conclusion reached is that oxygen is the most potent factor in the solution of lead. Whereas distilled water free from gases only dissolved 0·01 part per 100,000 in twenty-four hours at 24° C., and water under 1 atm. pressure of carbon dioxide 1·6 part, water under 1 atm. pressure of oxygen dissolved 12 parts. Of all the salts tried only ammonium nitrate had a solvent action of the same order as oxygen, and ammonium nitrate has to be present in a concentration approximating 25 parts per 100,000 before it is as corrosive as distilled water saturated with oxygen.

The function of carbon dioxide is a secondary but important one. In absence of carbon dioxide the action of oxygen ceases when the water is saturated with lead hydroxide, but in the simultaneous presence of carbon dioxide the lead is precipitated as carbonate or basic carbonate, and the action is continuous so long as any oxygen remains in solution. This is the mechanism of the familiar phenomenon known as the "erosion" of lead.

Waters containing the carbonates or bicarbonates of the alkali or alkaline earth metals give rise to a protective coating of lead carbonate, after the formation of which no more lead dissolves. If a phosphate is present, so little lead dissolves before the formation of a protective coating that it can only be detected by refined methods On the other hand, calcium sulphate affords no protection, and water containing calcium sulphate but no bicarbonate attacks lead vigorously.

G. C. J.

Estimation of Lithium in Mineral Waters. L. W. Winkler. (*Zeitsch. anal. Chem.*, 1913, **52**, 628-640.)—Isobutyl alcohol affords an excellent means of separating lithium chloride from the chlorides of sodium and potassium, and is much less objectionable to work with than amyl alcohol, which was recommended by Gooch. At room temperature 10 c.c. isobutyl alcohol dissolve only 0·5 mgrm. of potassium and sodium chloride, whereas amyl alcohol dissolves twice as much. From quantities of alkali chloride not exceeding 1 grm. the lithium may be separated as follows: The mixed chlorides are dried at 120° C., and when cold are extracted with successive small quantities of isobutyl alcohol (about 2 c.c. each time) delivered from a measuring cylinder, and the extracts decanted through a 4 cm. filter into a platinum crucible. About 10 c.c. of the alcohol should be used in all, and the amount measured. The filtrate is evaporated to dryness, the residue dissolved in a few drops of water, about five times its weight of ammonium sulphate added, the mixture dried on the water-bath and then in an oven the temperature of which is gradually raised until, at about 180° C., the contents of the crucible begin to emit fumes. The covered crucible is then heated over a small flame, which is subsequently raised until an incipient red heat is attained. The cover is removed once or twice until no further fumes are seen, and the crucible and lithium sulphate are then allowed to cool in the air and weighed. From the increase in weight of the crucible 0·5 mgrm. is deducted to correct for the solubility of the chlorides of sodium and potassium. In this way approximately 90 per cent. of the lithium is recovered. The residue from the treatment with isobutyl alcohol is dissolved in water, the solution acidified with hydrochloric acid, evaporated to dryness, dried at 120° C., re-extracted with isobutyl alcohol as before, the residue is dissolved as before, and the whole series of operations repeated a third time. The quantity of lithium sulphate obtained from the third extraction, after correcting for the solubility of sodium and potassium chloride, is usually of the order of 0·1 mgrm. The chlorides of calcium, magnesium, and aluminium are soluble in isobutyl alcohol, and must be absent from any saline residue submitted to the process.

The process as described can be applied to the mixture of alkali chlorides obtained in the ordinary course of a water analysis, but lithium will usually be under-estimated if lime salts have been eliminated in the usual manner. In a water with 100 degrees of hardness, approximately 10 per cent. of the lithium will be co-precipitated with the calcium as carbonate, and in a water of 10 degrees of hardness about 1 per cent. of the lithium will be lost. It is better to determine lithium in a separate portion of the sample. If the water has more than 100 degrees of hardness, it is first diluted. Waters less hard are not concentrated, but treated with excess of alkali hydroxide and carbonate to precipitate magnesium and calcium. A litre of the water is treated with these reagents, heated on the water-bath for an hour, allowed to cool, filtered, and the residue washed with water containing a little alkali carbonate and hydroxide. If the lime-hardness exceeds 10 degrees, the precipitate is dissolved in hydrochloric acid, the solution evaporated to dryness, the residue redissolved in a litre of water, and calcium and magnesium reprecipitated as before. The united filtrates are acidified with hydrochloric acid, treated with excess of barium chloride and a little nitric acid (to destroy organic matter), and evaporated to a pasty consistency. The lithium is next separated from the greater part of the other alkali chlorides present by treating the pasty mass with five times its own weight of absolute alcohol, which is decanted off through a filter. The residue is redissolved in water, the solution acidified with hydrochloric acid, evaporated to a paste, and the paste extracted with alcohol as before. The residue is again dissolved, and the series of operations repeated. The alcoholic filtrates are united, diluted with an equal bulk of water, and evaporated to dryness. The residue is dissolved in water acidulated with hydrochloric acid, and the solution, usually cloudy, is filtered and evaporated to dryness. The saline residue is freed from the last traces of calcium and magnesium by solution in 10 to 20 c.c. water, treatment with a few drops of a solution of alkali hydroxide and carbonate and filtration, washing with a dilute solution of the precipitant. The filtrate is acidified with hydrochloric acid, evaporated to dryness, and the residue redissolved in 10 to 20 c.c. water. The last trace of aluminium is eliminated by addition of a few drops of ammonia, heating to boiling, allowing to stand an hour, and filtration, washing with water containing a few drops of ammonia. A few drops of alkali hydroxide solution are added to the filtrate, which is next evaporated to half bulk to expel ammonia, as ammonium chloride increases the solubility of potassium and sodium chlorides in isobutyl alcohol. Finally, the solution is acidified with hydrochloric acid, evaporated to dryness, and the saline residue dried at 120° C. Usually it will weigh less than 1 grm., and the lithium is then separated as described in the opening paragraph; but if much more than 1 grm., the lithium should first be concentrated by treatment with alcohol. A single extraction with ten times its weight of alcohol and filtration through a plug of cotton-wool suffices at this stage. G. C. J.

Hydrolytic Action a Source of Error in the Estimation of Iodine and Bromine in Mineral Waters, etc. P. Kaschinsky. (*Zeitsch. angew. Chem.*, 1913, **26**, 492-494.)—Owing to the dissociation of magnesium halides according to the equation $MgX_2 + 2H_2O = Mg(OH)_2 + 2HX$, the whole of the iodine and almost all the bromine present as iodides and bromides in a mineral water may be lost when the water is evaporated and the residue ignited. It is essential that the magnesium salts be removed by precipitation with alkali hydroxide and subsequent filtration before the iodine and bromine are estimated. Potassium hydroxide is to be preferred to calcium hydroxide for the precipitation of the magnesium salts, as potassium chloride is less soluble than calcium chloride in the alcohol which is used in the process. W. P. S.

Estimation of Small Quantities of Hydrogen Sulphide in Natural Waters. L. W. Winkler. (*Zeitsch. anal. Chem.*, 1913, **52**, 641-645.)—The reagent employed for producing the coloration, the intensity of which is made the measure of the hydrogen sulphide present, is a solution of 10 grms. Rochelle salt, 10 grms. ammonium chloride, and 0·1 grm. lead acetate in 100 c.c. of 5 per cent. ammonia. The Rochelle salt and ammonium chloride prevent the precipitation of calcium carbonate and the hydroxides of magnesium and iron, and Rochelle salt also prevents the development of any interfering colour due to iron sulphide. The standard solution used for making the colour comparison is an approximately 0·1 per cent. solution of sodium sulphide, $Na_2S.9H_2O$, a salt which can now be obtained (from Kahlbaum) substantially pure. Whereas a dilute solution of sodium sulphide in distilled water suffers a loss of titer of 35 per cent. or more in twenty-four hours, a solution made up with a 50 per cent. solution of sodium nitrate instead of water loses its titer at less than one-third of this rate. Such a solution has been found useful, but it should be used as soon after preparation as possible.

A completely satisfactory determination of hydrogen sulphide in water can only be made at the source. The sodium sulphide is best conveyed to the point of collection of the sample as follows: About 0·1 grm. is accurately weighed into a weighing-bottle of not more than 1 c.c. capacity. The stopper is then greased, the bottle taken to the place of collection, and its contents dissolved in 100 c.c. of sodium nitrate solution rendered feebly alkaline with ammonia.

The water to be examined is collected in a bottle of known volume (about 100 c.c.), 5 c.c. of the lead reagent is added, the stopper inserted, and the contents shaken and then transferred to a 200 c.c. beaker. In a second beaker, 100 c.c. of distilled water and 5 c.c. of the lead reagent are mixed, and standard sodium sulphide solution is run in from a burette until the tint matches that of the sample under examination.

If the water to be examined is naturally coloured, the influence of this colour may be corrected for as follows: Some of the water is treated with the lead reagent placed in a stoppered bottle, which it should only half fill, and shaken at intervals for an hour or two. The lead sulphide at first formed is thus oxidised, and the resulting solution, identical with the original water in colour, and free from hydrogen sulphide, is then used instead of distilled water for the preparation of the standard of comparison.

G. C. J.

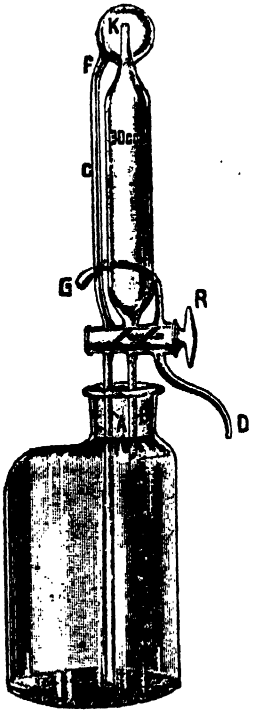
APPARATUS, ETC.

Use of Bomb Calorimeter for Elementary Analysis and Heat of Combustion. M. Diakow. (*Biochem. Zeitsch.*, 1913, **55**, 116-124.)—In the application of the bomb calorimeter to the study of metabolism, the oxidation of nitrogen to nitric or nitrous acid is a serious source of error. It is usual to correct for this by estimating the acid formed and assuming it to consist entirely of nitric acid. This assumption is only correct for ash-free substances; in other cases a direct estimation of nitric acid is necessary. Carbon, oxygen consumed, and nitrogen liberated, are estimated by the following method: The bomb is weighed before and after the introduction of oxygen, and after combustion a part of the gas is passed through a carbon dioxide absorption apparatus, and a part is collected and analysed, the carbon dioxide, nitrogen, and oxygen being estimated. From the total carbon dioxide and the percentage of carbon dioxide in the gaseous product, the total gaseous product, and hence also consumed oxygen and liberated nitrogen, can be calculated. In correcting for the "apparent nitrogen" in the compressed oxygen, it is necessary to bear in mind that this gas consists largely (76 per cent.) of argon.

E. W.

New Method and Apparatus for the Estimation of extremely small Quantities of Carbon Dioxide. Shiro Tashiro. (*Amer. J. Physiol.*, 1913, **32**, 137; through *Chem. Zentralbl.*, 1913, II., 978.)—The smallest quantity of pure carbon dioxide able to cause turbidity in a drop of known barium hydroxide content is first determined; the smallest quantity of the gas under investigation able to produce the same turbidity in a similar drop is then found. The apparatus consists of two chambers of known volume joined by a capillary tube provided with a stopcock; into the first a drop of the barium hydroxide solution is introduced through a capillary, and the gas sample, contained in the second chamber, is then forced through the stopcock until turbidity is produced. Details are to be found in the original paper.

O. E. M.



Automatic Pipette. P. Medinger. (*Chem. Zeit.*, 1913, **37**, 1107.)—The pipette illustrated differs from other pieces of apparatus designed to serve the same purpose in having a single cock, which by turning—always in the same direction—provides for filling the pipette, equalising the pressure within and without the apparatus, discharging the pipette, and, after recharging it, discharging the liquid which would otherwise be retained by capillarity in the discharging jet. With the cock in the position shown, the pipette is filled by means of a pressure ball attached to *G*. It is then given a quarter turn, whereby the bottle is put in connection with the atmosphere by means of a channel grooved in the body of the plug, which channel in this position of the plug connects *B* with *D*. Another quarter turn leads to the discharge of the contents of the pipette through *D*, and to the flow back of liquid from *K* to the bottle. Another half turn brings the cock once more to the filling position. The pipette is filled, and the next quarter turn of the cock not only equalises the pressure within and without the apparatus, but leads to the discharge of liquid in *D* by the escaping air. The receiver below *D* is then changed, and the cycle of operations repeated. When not in use, the plug is so turned that the horizontal channel is at the top connecting the pipette with *G*. The makers, Warmbrunn, Quilitz and Co., Berlin, deliver the apparatus with the plug of the cock constructed of glass, silver, or phosphorbronze.

G. C. J.

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REPORTS.

Report to the Local Government Board on Bacterial Food-Poisoning and Food Infections. W. G. Savage. (*Food Reports*, No. 18, 1913.)—This Report presents a summary of our present knowledge on the subject of bacterial food infections, and is a continuation of Dr. Ballard's review in the Annual Report of the Medical Officer of the Board for 1890. A memorandum issued by the Board, September, 1911, is again printed as Addendum B., p. 68, and Dr. Newsholme concludes that "Recent additions to our knowledge concerning the procedure to be followed in dealing with outbreaks of illness suspected of being due to the consumption of certain foods, tend to emphasise rather than to lessen the importance of inspection of meat at slaughter-houses, of the exercise of adequate control in regard to the suitability of places where food is prepared and stored, and of measures to secure cleanliness in all stages of food preparation." Cases of so-called "ptomaine-poisoning" are now generally recognised to result from the presence in the food materials consumed of certain pathogenic organisms (generally members of the Gaertner group), which are either living, capable of active multiplication, or which, although killed in the cooking, are nevertheless poisonous by means of the toxins contained in them. A large number of cases of food-poisoning are summarised, and it is concluded that for none of them was it definitely established that *B. proteus* was etiologically concerned, nor is the available evidence sufficient to establish that this organism is ever a cause of extensive outbreaks of food-poisoning. It still remains a matter of controversy as to how far the occurrence of putrefaction in meat may be a cause of even isolated cases of human disease, while for extensive widespread outbreaks it can certainly be excluded.

Section 5 deals fully with the Gaertner group in relation to food-poisoning, and the ease with which the toxins of this group may escape destruction when subjected to ordinary cooking temperatures is especially emphasised; nor are the ordinary methods of food preservation, such as smoking and pickling in brine, very efficacious for their destruction. The study of recorded outbreaks in this country shows in a considerable number of them that infection of the foodstuff may with great probability be accepted as taking place during or after the preparation of the animal for sale, although it is certainly probable that in other cases the food was contaminated because it was derived from an animal suffering from a general or local disease caused by one or other member of the true Gaertner group, animals used for food being not uncommonly affected by such organisms. The following three hypotheses are considered at some length: (*a*) That the Gaertner group bacilli which are the cause of the food-poisoning outbreak are of human origin, the meat being infected with pathogenic Gaertner bacilli from a human source—*i.e.*, a case of disease (paratyphoid fever) or a carrier case. (*b*) That the Gaertner group bacilli which set up the food-poisoning outbreaks are derived from ordinary fæcal infection of the food. (*c*) That food-poisoning outbreaks are due to infection of the food with virulent Gaertner group organisms (or other special bacilli) derived from animals which are either at the time suffering from disease due to Gaertner group bacilli or acting as carriers of these bacilli. In Dr. Savage's opinion this last view is the one which best explains all or most of the phenomena of food-poisoning. From the review of existing knowledge as given in the Report it is clear, the author says, that from the preventive standpoint food-poisoning cases must be looked upon as forming two groups: (*a*) those due to the consumption of meat derived from diseased animals; and (*b*) those due to the contamination of meat or other food with food-poisoning bacilli derived from sources other than the food itself. It is probable that this second group is considerably larger than the first. A discussion of various preventive measures suggested concludes the Report. A useful addendum consists of a tabulated list of seventy-nine British and forty-four Continental outbreaks dating from 1878 to 1911, in which the number of cases, deaths, kind of food eaten, bacteria isolated, etc., are recorded. A bibliography consisting of 155 British and foreign references to the literature is also included. H. F. E. H.

Report of the Government Chemist upon the Work of the Government Laboratory for the Year ended March 31, 1913, with Appendices. (*Issued as a Parliamentary Paper* [Cd. 7001], pp. 1-25.)—The total number of samples analysed and examined in the Government Laboratory during the year was 209,502; in addition to these, 145,257 samples were examined at the chemical stations in the provinces.

With a view of preventing or detecting the use of prohibited substances, 91 samples of finished beer from brewers' premises were specially examined as to the presence of saccharin, saponin, etc., but no deleterious or illegal substance was detected in any of the samples. As regards dilution of beer by retailers, out of 9,453 samples analysed, 581 were found to have been diluted. The dilution did not usually exceed 3 gallons of water to the barrel of 36 gallons, but occasionally was as high as 12 gallons to the barrel. The amount of alcohol was estimated in 356 samples of herb beers, beer substitutes, and temperance beverages. In 268 cases the quantity of alcohol was below 2 per cent. of proof spirit; 63 samples contained between 2 and 3 per cent., 15 between 3 and 5 per cent., whilst 10 samples described as ginger beer or herb beer contained from 5·7 to 11·5 per cent. of proof spirit. The number of samples of beer and brewing materials examined for the presence of arsenic was 815. Of these only 16 were found to contain arsenic in excess of the limits laid down by the Royal Commission on Arsenical Poisoning. Out of a total of 434 samples of malt and sugar, 2 samples contained excess of arsenic, the amount being $\frac{1}{50}$ gr. per pound in one case, and $\frac{1}{80}$ gr. in the other. The limit was exceeded in 5 out of 242 samples of beer and wort, but in none of the samples did the amount exceed $\frac{1}{45}$ gr. per gallon. Nine of the miscellaneous samples examined contained an excess of arsenic, but, with one exception, it was of a trifling character. During the year 6 samples purchased as cider were found to be artificially prepared liquids; of 262 samples of imported cider 2 were found to be factitious. Many non-alcoholic ciders, so-called, are entirely free from fermented apple-juice, and consist of solutions of sugar which have been aerated, flavoured, and coloured. The use of any name for such beverages, which suggests that they consist of fermented apple-juice or cider, is an infringement of the Merchandise Marks Act. Twenty-three samples of liqueur chocolates were examined for the presence of alcohol; 6 contained from 5 to 8 per cent. of proof spirit, and in 2 cases the amounts were 23 and 27·4 per cent. The average percentage of proof spirit found in 269 samples of imported liqueur chocolates was 5·4, the highest amount being 21·9 per cent.

The number of samples examined for the Board of Agriculture and Fisheries, the Board of Agriculture for Scotland, and the Department of Agriculture and Technical Instruction for Ireland, was 2,944.

Imported Milk and Cream.—Of the 73 samples of condensed sweetened and unsweetened milk, 65 were condensed whole milk, and 8 condensed skim milk; all the cases were properly marked. The percentage of fat in the samples of whole milk ranged from 8·0 to 11·3, and in no case was there evidence that the original milk contained less than 3 per cent. of fat. Of the 64 samples of dried milk analysed, 15 were whole milk powders, 5 had been prepared from impoverished milk, and 41 from skimmed milk. Two of the samples consisted of milk proteins and 1 of lactose only. The range of fat in the cream imported from France was 40·4 to 65·8 per cent., and in that from Norway 18·3 to 32·1 per cent.

Imported Butter.—Out of 1,309 samples, 13 contained between 16 and 17 per cent. of water, and 6 samples more than 17 per cent. All the samples were free from foreign fats.

Imported Margarine.—None of the 598 samples examined contained more than 10 per cent. of butter fat, but water in excess of the legal limit was found in 5 cases. The samples were all free from paraffin wax.

Imported Cheese.—The 65 samples analysed were free from fat other than milk fat, but in several cases the amount of fat was very small, indicating that the cheeses had been prepared from skimmed or separated milk.

Eighty-three samples of sheep-dips were received for examination; 12 were found to be deficient in active ingredients, and in 42 cases the formulæ required alteration to insure an efficient dip.

Samples of potatoes grown on experimental plots which had been treated with different copper fungicides were examined with the view of ascertaining whether the tubers had absorbed copper. No evidence of absorption was obtained.

In view of the statements to the effect that imported butter frequently contains considerable quantities of non-fatty milk solids, the curdy matter was estimated in 366 samples, representing practically every class of imported butter. The results obtained showed that the amount of curd in foreign butter is not excessive, and is quite comparable with the quantity which occurs in well-made butter in this country.

All the samples of milk, cream, and butter were tested for the presence of preservatives. The samples of whole milk imported in churns, and of sterilised milk, sterilised cream, and condensed milk, were free from preservatives. One sample of milk powder contained boric acid. The creams, when preserved, contained boric acid only, the amount of this varying from 0·12 to 0·46 per cent. All the samples of cream imported since October 1, 1912, complied with the new regulations which then came into operation. Boric acid was also the only preservative found in imported butters. The butters imported from Denmark, Russia, Norway, and Sweden were free from boric acid, whilst over 70 per cent. of those from France, Argentina, Australia, and New Zealand contained this preservative.

Fifty-eight glazes from various works where lead-poisoning had occurred were analysed to determine both the total amount of lead and the amount of soluble lead present. In most of these cases almost the whole of the lead, frequently amounting to 30 per cent. of lead monoxide, was found to be readily soluble in hydrochloric acid of a strength equal to that usually present in the gastric juices. "Leadless" glazed pieces of pottery were also examined, and in only a few cases was the amount of lead found in the glaze of the finished ware so great as to leave no doubt that ordinary lead glaze had been used in their manufacture. The permissible quantity of lead monoxide in a leadless glaze has been fixed at 1 per cent. to allow a margin for the small quantities of lead which may be picked up from the grinding vessels and dipping-tubs.

Ninety-two samples were referred to the Laboratory by magistrates under the Sale of Food and Drugs Acts; these included 74 samples of milk, 8 of butter, 2 each of ginger beer and vinegar, and 1 each of brandy, cream, cream of tartar, flour, rice, and whisky. In the case of 1 sample of milk in which decomposition had proceeded to an unusual degree, no certificate was issued. The results of the analyses differ from those of the public analyst in 5 cases. The following are the particulars of the more important cases:

Butter.—The results of the examination were in agreement with those of the public analyst in the cases of 2 samples of butter alleged to contain an excess of water, and in the case of a sample of butter containing an excess of boric acid. In 3 out of 5 cases in which foreign fat was alleged to be present the results of the public analyst were confirmed, but in the remaining 2 cases, although the butter was of abnormal character, no evidence of the presence of foreign fat was discovered.

Ginger Beer.—Lead to the extent of 8·3 and 6·5 grains per gallon was found in 2 samples in which it was stated to be present by the public analyst.

Vinegar.—One sample was found to contain 0·78 grain of copper per pint, and the other to be deficient in acetic acid, as alleged.

Brandy.—The public analyst reported that the sample contained spirit other than that derived from the grape, and this was confirmed, the sample being deficient in the characteristic constituents of brandy to such an extent as to indicate the presence of a large proportion of neutral spirit.

Cream of Tartar.—The allegation that the sample contained a considerable quantity of lead could not be confirmed.

Flour.—The sample contained nitrites, as alleged.

In regard to the samples of whisky alleged to be deficient in strength, of rice alleged to be faced with talc, and of cream alleged to be preserved with boric acid, the results agreed with those of the public analyst.

Milk.—In the 73 cases in which certificates were issued, the charges were as follows: In 33, added water; 26, abstraction of fat; 3, added water and abstraction of fat; 2, added water and colouring matter; 1, added water and boric acid; 5, boric acid; 2, other preservatives; and in 1 an excessive quantity of dirt. In connection with experiments and investigations arising out of the examination of the samples referred to under the Food and Drugs Act, 192 samples were analysed in order to obtain data for the purpose of comparison.

The number of samples submitted by the Board of Agriculture and Fisheries under the Fertilisers and Feeding Stuffs Act was 25, consisting of 15 fertilisers and 10 feeding stuffs. The fertilisers comprised samples of basic slag, various potato and root manures, wool waste, hoof and horn meal, grass manure, guano, fish manure, and sulphate of potash. A sample described as "dissolved bone" consisted mainly of mineral phosphates mixed with nitrates. The feeding stuffs examined consisted of samples of bran, barley meal, poultry food, and various feeding and cake meals. An article sold as barley meal contained a considerable proportion of ground weed seeds and seed husks; another sample of barley meal contained over 3 per cent. of sand. Several of the feeding meals and cakes were deficient in oil, and others contained sand and earthy matter in excess.

Details as to the numbers of various substances examined in the Laboratory and in the provincial stations during the year are given in the Appendices to the Report.

W. P. S.

Royal Commission on Sewage Disposal: Appendix to Eighth Report, March, 1913. G. McGowan, C. C. Frye, and G. B. Kershaw.—This Report contains the results of analysis and of physical observation of streams receiving sewages and effluents during the years 1909-12, together with notes on the tests in use and a description of the standard tests selected by the Commission for judging the quality of an effluent in relation to the stream into which it is discharged. These standard tests are two—namely, the dissolved oxygen absorption and the determination of suspended solids. The description of the standard conditions for carrying out these two tests form the items of greatest analytical interest in the Report.

Dissolved Oxygen Absorption in Five Days.—The principle followed is that of Winkler, with Rideal and Stewart's modifications (see ANALYST, 1901, **26**, 141). The sample of effluent in its bottle is well shaken for a half minute in order to mix any deposited solids and to effect fairly complete aeration. About 300 c.c. will be required, and this volume is accurately diluted with four times its volume of well aerated tap-water at 65° F. The dilution is gently mixed and poured quietly into four bottles, also at 65° F., the capacity of each being about 350 c.c., and being accurately known. The bottles are allowed to stand full to the mouth for five minutes and then stoppered. Two bottles are placed in an incubator at 65° F. for five days, and in the other two the amount of dissolved oxygen is determined at once, as follows: 0·9 c.c. of sulphuric acid is added and 1 to 2 c.c. of $\frac{N}{8}$ permanganate, and the contents mixed and allowed to stand for twenty minutes. The authors found that this length of time was necessary in order to oxidise any nitrites. Then the excess of permanganate is destroyed by the addition of about 1 c.c. of 2 per cent. potassium oxalate, and when the liquid has become colourless 1 c.c. of 33 per cent. manganous chloride is run into the bottom, and immediately afterwards 4 c.c. of alkaline iodide solution (70 per cent. potash and 10 per cent. potassium iodide) are added. The bottle is then inverted once or twice, allowed to stand for a few minutes, inverted again, and then allowed to settle. Five c.c. of hydrochloric acid are added, and the bottle allowed to stand in the dark for five to ten minutes with occasional rotation. Twenty c.c. of the liquid are pipetted out and thrown away, and the remainder titrated with $\frac{N}{0}$ thiosulphate, using clear starch solution as an indicator. The result is calculated into parts of dissolved oxygen per 100,000 parts of the mixture. The second bottle is treated in the same way, and the mean of the two results is taken. At the end of five days the dissolved oxygen in the bottles in the incubator is determined, and the mean of the results subtracted from the first mean gives the amount of dissolved oxygen absorbed by the mixture during the five days. This result multiplied by 5 gives the amount of dissolved oxygen absorbed by 100,000 parts of the effluent. The Commission has fixed a standard of 2 parts per 100,000 for the dissolved oxygen absorption.

For tank effluents the dilution should be 1 of effluent to 49 of tap-water, and for sewages, 1 volume to 99 volumes, the object being to have about half the dissolved oxygen in the mixture left unabsorbed at the end of the test.

As samples have usually to be sent from the outfall to a laboratory, and as it is desirable that results from different places should be comparable, it is recommended that all samples should be allowed to stand at 65° F. for a uniform time of forty-eight hours after sampling before carrying out the test. If it is not possible to commence the test at the end of that time, the sample should be kept on ice for any further period.

Solids in Suspension.—These are determined by filtering a known volume of the effluent through asbestos in a Gooch crucible. The crucible used should be about 1¾ inches high and 1½ inches in diameter at the top, and its weight should not exceed 20 grms. Asbestos floss is used, consisting of silky fibres about 2 cms. long, and is treated with acid to free it from iron before use. Some of the asbestos is placed in the crucible supported in a pressure filter-flask, and is pressed down with a flattened glass rod to form a layer about 0·2 cm. thick, a stream of water being kept running through the crucible. When all loose particles of asbestos have been washed away, the crucible is washed with distilled water, dried at 105° C., and ignited and weighed until constant.

In the case of average effluents, 200 c.c. of the shaken sample are poured into a measuring-flask for the estimation. The asbestos is moistened with a small quantity, then the pump is turned on so as to produce a reduced pressure of not more than 100 mm., and the remainder of the quantity run through gradually. If the filtrate should contain any deposited solids, a new estimation must be begun. Then the crucible is washed with distilled water, dried at 105° C., and weighed once every twenty-four hours until constant to within 0·3 mgrm. The increase in weight gives the solids in suspension. For this result, the Committee has fixed a standard of 3 parts per 100,000.

In the case of sewages and tank effluents, 25 c.c. of an average sewage and 50 c.c. of an average tank effluent should be taken. The authors express the opinion that the Gooch filtrates from these samples appear to contain about 3 or 4 parts of colloids.

The same directions as to the time interval between the making of the test and the taking of the sample are given as for the dissolved oxygen absorption test; in fact, both tests are made on the same bottle of sample.

River Waters.—A large number of physical observations were made upon the condition of the water in many rivers, and a full chemical analysis of samples representing the various conditions was undertaken. It was found that the results of the dissolved oxygen absorption test agreed best with the conditions observed. The figures obtained with this test corresponding to the various states of the river were as follow:

Condition of Reach of River.	Average Dissolved Oxygen Absorption.	
Very clean	0·10	
Clean	0·20	
Fairly clean	0·27	0·30
Moderately clean	0·31	
Doubtful	0·48	
Bad	0·97	

From these results the authors have adopted the figure of 0·40 part per 100,000 as the "limiting figure" for the dissolved oxygen absorption of a river water, if it is to be considered as in a satisfactory condition and as not exhibiting signs of pollution.

The Report also gives tables showing the chemical composition of the mud, and of various algæ and fresh-water plants found in rivers, for the analysis of which the ordinary methods were found suitable. J. H. J.

Report of the Connecticut Agricultural Experiment Station for the Year 1912. (Public Document No. 24, pp. 1-531.)—The work of the chemical, botanical, entomological, forestry, plant-breeding, and protein research departments of the Experiment Station during the year 1912 is recorded in the present report. Seven hundred and eighty samples of fertilisers and manures and 273 samples of feeding-stuffs were analysed, the results obtained being given in tabular form. The question of valuation is also considered in the case of the fertilisers, etc., and figures are given in most cases. The valuation of a fertiliser is the result obtained by calculating the retail cash cost at freight centres of an amount of nitrogen, phosphoric acid, and potash in high-grade materials equal to the amount contained in one ton of the fertiliser.

The variations in the net weight of certain classes of foods sold in package form were also investigated, about 2,000 packages, representing 150 brands of about 75 kinds of foods, being weighed. A definite weight was claimed on 594 of the packages examined, and in 77 cases the weight was found to be deficient.

The work of the botanical department included experiments on the effect of fertilisers on growth, yield, etc., of trees; the effect of various sprays; the study of the nature and spread of the chestnut blight; seed testing, etc. The entomological department has determined the life-histories of three little-known and destructive insects and the means for combating these and other pests. The protein research department is occupied in determining the nutritive effect of the separate proteins of foods. W. P. S.

REVIEWS.

THE NATIONAL PHYSICAL LABORATORY. COLLECTED RESEARCHES. Vol. x. 1913.

This volume of 253 pages, quarto, contains eleven papers on a variety of subjects, reprinted from various journals, reports, etc.

The first paper is the "Tenth Report to the Alloys Research Committee of the Institution of Mechanical Engineers," reprinted from the *Proc. Inst. Mech. Eng.*, April and May, 1912, and is an exhaustive study of the constitution, mechanical properties, and micro-structure of the alloys of aluminium and zinc, by Dr. W. Rosenhain and Mr. S. L. Archbutt. The authors conclude that the alloy containing 20 per cent. of zinc is the most generally useful of the simple binary alloys of these metals, and that those containing from 15 to 25 per cent. of zinc are better deserving of notice than the, at present, more widely used alloys containing less than 15 per cent. of zinc. When prepared from the pure metals, these alloys do not show the tendency to crack on cooling, or the brittleness or liability to rapid corrosion, which many of the commercially made alloys of this class have shown, and which the authors attribute to the presence in the latter of impurities. They can be rolled into bars and drawn into wire of considerable tensile strength, increased by the addition of a small percentage of copper, but the strength rapidly falls off with rise of temperature. The industrial alloys of this class containing less than 40 per cent. of zinc are shown to be homogeneous solid solutions. This Report will take its place among the classical researches on binary alloys, and will be found of value by all metallurgical analysts.

The second and third papers are mainly of theoretical interest. The first of them, reprinted from the *Journal of the Institute of Metals*, No. 2, vol. viii., 1912, contains an experimental research by Dr. Rosenhain and Mr. D. Ewen on "Intercrystalline Cohesion in Metals," which they believe to be due to the existence between the crystals of a cementing film of metal in the amorphous form discovered by Beilby. In this paper a new method of developing the micro-structure of metals by "vacuum etching" is described, and illustrated by some highly interesting and beautiful photographs. In the third paper, reprinted from the "Carnegie Scholarship Memoirs of the Iron and Steel Institute," vol. iv., 1912, Mr. J. C. W. Humphreys shows that intercrystalline brittleness may be caused in iron by annealing in a slightly oxidising atmosphere, and he describes experiments in support of his theory that this is due to the existence of an oxide of iron which is soluble in γ iron, but not in α iron, and is therefore rejected to the boundaries of the crystals on cooling. In an appendix to this paper some electric-resistance furnaces are described, of simple construction, for annealing and melting metals *in vacuo* in the laboratory.

Papers on "The Measurements and Notes on the Visibility of Point Sources of

Light," by Messrs. Paterson and Dudding; on "Tests of Tungsten Filament Glow Lamps, made for the Engineering Standards Committee," by Dr. Glazebrook and Mr. Paterson; on "The Transmission of Visible Light by Photographic Lenses," by Mr. R. W. Cheshire; on "The Shape of Scales required for Reflecting Instruments with Concave Mirrors," by Mr. E. H. Rayner; on "The Ideal Photographic Lens," by Mr. T. Smith; and on "The Results of Some Tests of Photographic Shutters," by the same author, are not of special interest to the readers of this journal.

In the paper on "The Testing of Ebonite for Electrical Purposes," by Messrs. Paterson, Rayner, and Kinnes, reprinted from the *J. Inst. Elect. Eng.*, Part 217, vol. l., 1913, there are several points of interest to analysts. It is shown that ebonite made from Para rubber and sulphur only has a far higher electric strength than that made from any other kind of rubber, and that all mineral admixtures and other adulterants reduce the strength; also, that whilst the electric strength can only be ascertained by an electric test which is costly to carry out, information of value can be obtained by a determination of the specific gravity and by a yield test which the authors describe, and which depends upon the bending of a weighted cantilever of the material at 70° C. It is stated that all ebonites, without exception, suffer from the defect of exuding small drops of acid upon the surface under the influence of light.

In "An Investigation into the Inflammability of Flake Charcoal, Decayed Wood, and Insulating Papers, made for Lloyd's Register of Shipping," by Dr. Rosenhain and Mr. W. Gemmell, it is shown that flake charcoal used in the insulating partitions of refrigerating chambers does not heat to a dangerous extent as a result of the absorption of gases into the pores of the charcoal, but that when subjected to a current of air such charcoal commences to burn at comparatively low temperatures. The following table contains the results of the experiments made:

Material.	Rate of Air-Supply in Cubic Feet per Hour.	Temperature at which Ignition occurs.
		° C.
Flake charcoal...	1·2	111
" "	5·0	110
" "	62·0	96
Decayed wood, No. 1...	5·0	180
" " No. 2...	10·0	178
Brown unvarnished paper	5·0	195
Black varnished paper, No. 2	5·0	216
"Ruberoid"	5·0	228

If the air contain as little as 5 per cent. of sulphur dioxide, spontaneous ignition of the charcoal may take place without the supply of any extraneous heat, and therefore the fumigation with SO_2 of ships having charcoal-fitted bulkheads is a dangerous proceeding.

L. Archbutt.

DAIRY TECHNOLOGY. By C. LARSEN, M.S.A., and W. WHITE, B.S. New York: John Wiley and Sons. 1913. Pp. 298. $1.50 net.

This volume, which is decidedly American, and deals with the technique of dairy work principally and, in fact, almost entirely, from the American point of view, is very largely a compilation from many sources, and particularly from the numerous and excellent bulletins of the U.S. Department of Agriculture and of various State Experiment Stations.

Though a compilation, it has the rare merit of being compiled by those who are really conversant with their subject, and, consequently, selection has been judicious, and facts and details seem to be in all cases reliable. It is, indeed, quite refreshing to find at the outset a really good average composition of milk. A very large number of subjects are compressed into a rather small space, and the information is therefore somewhat telegraphic, and might have been a little more diffuse than it is.

There is an excellent synopsis of methods which have been used to improve the milk-supply of America, and, naturally, full details of the working of certified milk depots are given.

The *pros* and *cons* of pasteurised milk are carefully debated in a chapter which is quite up to date, and properly completed by a discussion on "cost." The details of the "handling" of milk in American cities are of a practical character.

One chapter is devoted to some curious methods of graphic calculation of milk mixtures for the purpose of "standardisation" of milk. Our American cousins seem to have grasped the fact that milk should be paid for according to quality, and that if a low-grade milk be required, it is quite legitimate to bring down a higher-grade quality to meet the requirement. Such ideas, however, are quite heretical in England.

In the second part of the book will be found many really useful little details and hints in connection with dairy products which should be useful to those who handle such.

A good deal of space is devoted to ice-cream, and there is probably no such elaborate treatment in any dairy work outside America. A full description of all the various trade products and nomenclature is given, but the manufacture of ice-cream is not of the same extensive nature in England, where it seems to be chiefly in the hands of itinerant vendors from a southern clime.

The chapters on milk-sugar, casein, fermented milks (which for some extraordinary reason include clotted cream), condensed and dried milks, etc., are all probably better than anything yet published in any dairy work. The processes given are all "live" processes, as far as the trade will allow its secrets to be published.

The final chapter on "Oleomargarine" is a little disappointing, but in view of the rapid changes in the composition of this product, it would be clearly impossible to keep descriptions up to date.

The book is well printed, and there are many illustrations, taken largely from trade journals and official bulletins. It is, however, unfortunate that practically no allusion is made to them in the text, and descriptions would have largely increased their utility.

The work will be probably of little interest to the general analyst, but should prove useful to those whose work lies chiefly in the dairy world or comes into contact with it.

CECIL REVIS.

FARINES, FÉCULES ET AMIDONS. Par MARCEL ARPIN. Paris: Librarie Polytechnique Béranger, 15, Rue des Saints-Pères. Price 6 f.

M. Arpin has long been well known as Chemical Expert to the Tribunal of the Seine, and also Technical Adviser to the Syndicate of Baking of Paris and to the General Syndicate of French Bakers. The present work of 190 pages is a practical treatise on the analysis of the substances indicated in the title. Among bodies dealt with are wheaten flour, the flour of rye, barley, and other grains. There is next given a description of bread and bread analysis, and the composition of *fleurages*, or accessory materials of the bakery. Chapters follow dealing with pastries, biscuits, and spiced bread. Regulations dealing with army and navy bread are also included.

As an example of the mode of treatment, it may be mentioned that the section on the analysis of wheaten flour contains directions for the determination of moisture, gluten, water of hydration of gluten, fatty matters, acidity, and ash. Microscopic analysis and the introduction of mineral matters into the flour are also dealt with. Then follows an account of the interpretation of the results of analysis.

One of the most striking features of the book is the minute attention to the details of analytical methods shown throughout. Thus, in giving directions for the performance of a gluten estimation, stress is first laid on the fact that numerous causes influence the constancy of the results. Thus, they are affected by the composition of the water employed, the temperature of the water, the time the dough is allowed to stand before the gluten extraction, and the duration of the process of extraction.

In making the estimation the author prefers to use water containing about 0·1 grm. of total lime per litre, of which from eight to nine-tenths should be in the form of carbonate. Directions are therefore given to take 0·1 grm. of quicklime prepared from marble, to grind this finely, and make up to a litre. This water should be used at a temperature between 15° and 17°; 33·33 grms. of flour are to be used for each determination and mixed with 17 c.c. of water. This is made into a perfectly smooth dough, and then, for reasons given by the author, the washing out of the gluten is commenced immediately. The most important of these reasons is that the hydration of gluten proceeds for a considerable time, attaining its maximum at the end of about four hours. Therefore, logically, the dough should be allowed to stand this length of time before the extraction is made. As it is inconvenient to wait the whole time, the author prefers, therefore, not to wait at all. In support of this decision be points out that this manner of procedure possesses the great advantage of indicating the quality of the flour, for the gluten which does so is all the better for the baker which absorbs the greatest quantity of water in the least time, and which is the most elastic, homogeneous, and cohesive. Minute directions follow for the manipulation of the dough during washing and freeing from surplus water, which it is stated should altogether occupy twelve minutes. The gluten is then weighed in the wet state, placed on a nickel plate, which has been treated with a trace of vaseline and dried at 105° C. for about twenty minutes. A number of incisions is then made in the mass of gluten so as to facilitate the escape of moisture, and the drying is continued for twelve hours. The gluten is then again weighed. Other analytical operations are described with the same minutiæ of detail.

One cannot altogether agree with M. Arpin in his reasons for immediately washing out the gluten on preparing the dough. The result most instructive to the baker would be obtained by allowing the dough to remain as long as it usually does before going into the oven, since that is the crucial time when the effects of the gluten are of service. The dough of Durum wheat flour would yield very little gluten on being first made; and yet after softening and hydration a considerable quantity of gluten is obtained from such flour. The present writer has obtained some very interesting gluten results from washing fermented dough taken from loaves just ready to be put in the oven.

Elsewhere reference has been made to the fact that in an abstract of Arpin's method of gluten estimation, appearing in the *Journal of the Society of Chemical Industry* (1902, **21**, 1417), it is mentioned that, "although there are numerous sources of error in the separation of gluten, the valuation of a flour on the basis of its content in moist gluten has not yet been suppressed." It is significant that, in 1913, M. Arpin, with his extensive experience, still adheres to the gluten-washing process, and gives no methods for determining protein, nitrogen, or gliadin, or glutenin in a flour. With all its possibilities of error, the extraction of gluten, not merely for its weight percentages, but largely because of the information given by its physical appearance and character, is a test which affords most valuable guidance to both the miller and baker.

M. Arpin's book will form a valuable addition to the library of the food analyst.

WILLIAM JAGO.

THE CHEMISTRY OF RUBBER. BY B. D. PORRITT, B.Sc., F.I.C. Pp. 87 + Contents + Bibliography + Index. London: Gurney and Jackson. 1913. Price 1s. 6d.

This little work is one of a series of Chemical Monographs edited by Dr. A. C. Cumming, and intended primarily for advanced and honours students. It is divided into six sections, of which those on Constitution and Derivatives (II.) and on Theories of Vulcanisation (IV.) are by Mr. F. D. Miles, B.Sc., A.R.C.Sc. The other sections are as follows: I. The Properties of Crude Rubber; III. Methods of Vulcanisation; V. Waste Rubber and its Utilisation; VI. Synthetic Caoutchouc. Mr. Porritt has set himself the task of giving a concise and accurate account of the chemistry of rubber within a very brief compass, and he has accomplished this task by adhering closely to the subject matter as indicated by the title. The monograph should be useful for the class of reader for whom it is intended. The work has been done on scholarly and scientific lines, although slips are noticeable here and there. Thus, on page 2 there is a reference (172) to the ANALYST which is not correct; and on page 3 we read *Kickxia* where *Castilloa* was, no doubt, intended. P. SCHIDROWITZ.

THE VOLATILE OILS. By E. GILDEMEISTER and FR. HOFFMANN. Second edition by E. Gildemeister, translated by Edward Kremers. Vol. I. With two maps and numerous illustrations. 8vo. Pp. xii + 677. London: Longmans, Green and Co. 1913. Price 20s. net.

The work by Gildemeister and Hoffmann may be regarded as a classic in the special field of literature which it represents. On account of its authoritative character as a source of information concerning the large and important group of natural products known as the volatile or essential oils, it may be assumed that it is now quite familiar to all who are interested in the subject. It may, indeed, be considered to have become indispensable as a work of reference, not only for the analyst or consulting chemist, but also for those engaged in the strictly scientific study of the essential oils and their numerous constituents.

Since the first appearance of the English version of this work thirteen years have elapsed, and during this period great advances have been made in the department of knowledge of which it treats. Many new volatile oils have in the meantime been obtained and described, and the constituents of many others have been more completely determined and characterised, whilst the methods for their examination have been correspondingly improved. It is therefore not surprising that the present revision of the work has necessitated a very considerable expansion of its contents. The extent of this development is evident from the fact that, although the portion of the first edition which treated of the theoretical principles of steam distillation, as applied to the volatile oils, has been issued as a separate treatise, the material comprising the principal part of the work could now only be adequately considered by its division into three volumes.

In order to indicate the scope of the volume now under notice, the following brief survey of its contents may be given. It includes: I. An historical introduction (241 pages); II. a chapter on the production of flower perfumes by extraction, enfleurage, and maceration (23 pages); III. an enumeration and description of the principal constituents of volatile oils and natural and artificial perfumes (280 pages). These constituents comprise such diverse compounds as the hydrocarbons, aliphatic, aromatic, and alicyclic, together with the sesquiterpenes; alcohols, aldehydes, ketones, phenols and phenol ethers, acids, esters, lactones, oxides, and compounds containing nitrogen and sulphur. IV. The examination of volatile oils (64 pages). This last section, which is particularly important for the analyst and for all who require to ascertain the purity and quality of essential oils, includes the determination of their physical properties and the chemical methods of examination. It is accompanied by very complete tables for computing the percentage of alcohols and the ester value, a second copy of the tables having also been added in such a form as to permit of their separate use in the laboratory. An excellent index is likewise provided.

It would be difficult to consider in further detail the contents of this very comprehensive work. Its completeness and accuracy render it in all respects a thoroughly trustworthy source of information on the subjects of which it treats, and it therefore merits the highest commendation. The translation of such a work from the original German into English could by no means have been an easy task, and the admirable manner in which this has been accomplished is also deserving of recognition. F. B. POWER.

TABLES ANNUELLES DE CONSTANTES ET DONNÉES NUMÉRIQUES. Vol. II., Année 1911. Gauthier, Villars, Paris; Akademische Verlagsgesellschaft m. b. H., Leipzig; J. and A. Churchill, London; University of Chicago Press, Chicago. 1913. xl + 759. Cloth. Price 24s. net.

The first volume of this very important annual was reviewed in the ANALYST for 1912 (**37**, 483), and the new volume, giving constants determined in 1911, has recently made its appearance; it is up to the high level of its predecessor. The list of compilers contains the following well-known English names: M. P. Appleby, S. L. Archbutt, A. W. Porter, L. J. Spencer, and N. T. M. Wilsmore; and as their foreign colleagues are of equal rank, one is assured that the work is well looked after. These compilers have been assisted by a large staff of abstractors, the British Empire and Germany being prominent in having furnished the larger number.

After introductory notes explaining the use of the tables, one encounters an index of chapters (1 page) and a table of contents (18 pages), so that even without an index information given about some particular property is easily obtained. It is interesting to note the extent to which different branches of science engage the attention of chemists, and the influence of fashion, which is certainly not confined to articles of attire. Thus "colloids" and "adsorption" occupy 30 pages in Volume I.; in Volume II. the space has grown to 44 pages, whilst, on the other hand, crystallography and mineralogy are reduced from 52 to 32 pages.

The printing and get-up generally are excellent. The tables should be readily accessible to all chemists, and should be found in every library attached to a chemical laboratory having any pretensions. J. T. HEWITT.

DECEMBER, 1913. Vol. XXXVIII., No. 453.

THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

An ordinary meeting of the Society was held on Wednesday evening, November 5, in the Chemical Society's Rooms, Burlington House.

The President, Mr. L. Archbutt, F.I.C., occupied the chair.

The minutes of the previous ordinary meeting were read and confirmed.

Certificates of proposal for election to membership in favour of Messrs. J. J. Eastick, A.R.S.M., F.I.C., and J. F. Millar were read for the second time; and certificates in favour of Messrs. Alan Milsom Bailey, A.I.C., Lanhill, Chippenham, Wilts, Second Assistant Government Analyst, Straits Settlements, Singapore; Arthur Leslie Barton, 20, Hotham Road, Putney Common, S.W., Chief Chemist and Bacteriologist to the Belgravia Dairy Company, Ltd.; Thomas Sidney Haines, A.I.C., 24, Woodfield Avenue, Ealing, Assistant to Mr. Cyril Dickinson, B.Sc., F.I.C.; and Arthur George Abraham Miller, 1, Pretoria Terrace, Waltham Cross, Herts, Lecturer in Chemistry and Physics at the Sir George Williams College, Tottenham Court Road, London, were read for the first time.

Messrs. C. W. Birch, U. R. Evans, B.A., and H. W. Gill, B.Sc., A.I.C., were elected members of the Society.

The following papers were read: "The Preparation of Rubber for Analysis," by L. Archbutt, F.I.C.; "The Examination of Commercial Gelatins in Reference to their Suitability for Paper-Making," by R. W. Sindall and W. Bacon, B.Sc., F.I.C.; "Some Experiments on Chlorine Compounds of Ethane and Ethylene, with Special Reference to their Application to Analytical Chemistry," by L. Gowing-Scopes; and "The Detection and Estimation of Benzoic Acid in Milk and Cream," by Edward Hinks, B.Sc., F.I.C.

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THE PREPARATION OF RUBBER FOR ANALYSIS.

By L. ARCHBUTT, F.I.C.

(Read at the Meeting, November 5, 1913.)

THE preparation of rubber for analysis is a matter which is not very satisfactorily dealt with by the textbooks, and one does not find a machine for the purpose included in the catalogues issued by the dealers in laboratory apparatus. Weber suggests grinding the sample between a pair of mixing rollers; but the laboratory type of machine which he illustrates is power-driven, and would be heavy and expensive.

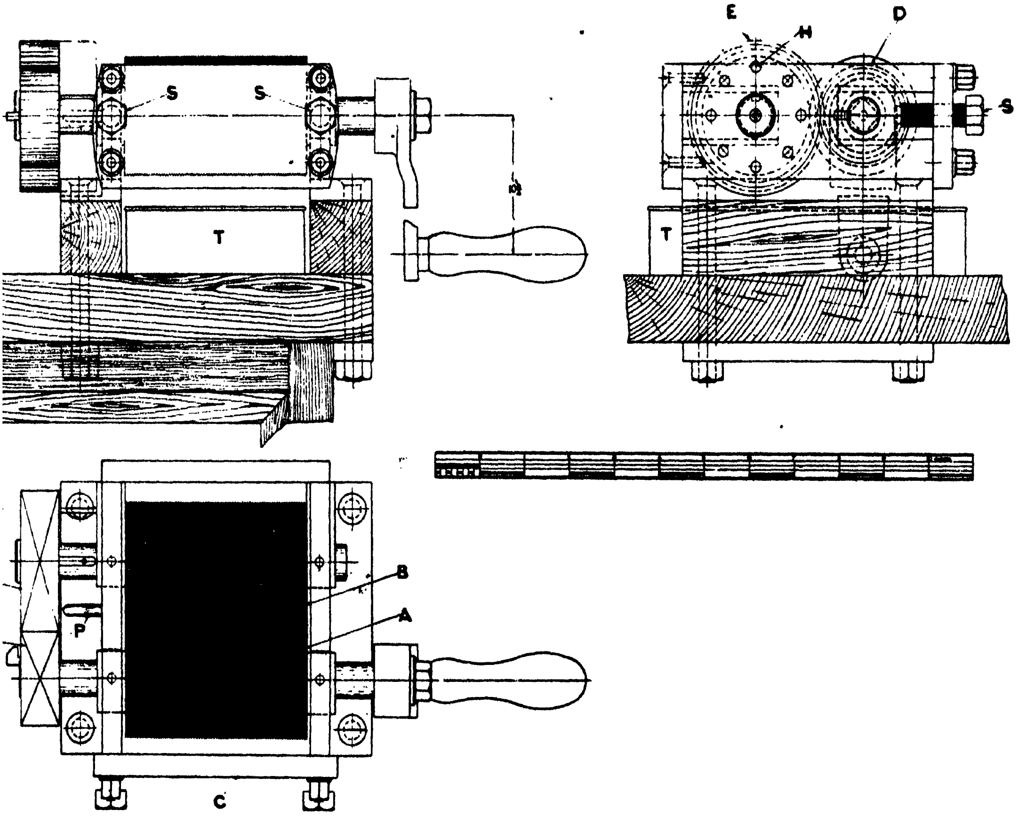
A method commonly employed, and one that answers well enough for an occasional sample, is to reduce the rubber to powder by rubbing on a coarse file or rasp; but this becomes very tedious when many samples have to be dealt with. Moreover, some soft rubbers, very small tubes, and materials such as the insulating coatings of wires, cannot be dealt with in that way; and even with fairly hard rubbers it takes a long time to rasp any considerable quantity.

In the course of my inquiries for a suitable machine I applied to Dr. Schidrowitz, who informed me that he had found it convenient to pass the rubber first through a pair of finely-grooved rollers, and afterwards to grind the partially broken-up material carefully in an ordinary coffee-mill. Acting upon this suggestion, a small experimental machine was made for me in the Midland Railway Works, from which, the machine, which I exhibit this evening, has been developed. By the simple device of mounting the rollers so as to work either in gear or with one roller fixed, we have found it possible to reduce all the rubber samples we have had to deal with to powder without any other appliance or treatment. The machine is easily operated by hand, quite simple, and can be constructed at the cost of a few pounds.

Referring to the figures, *A* and *B* are two solid grooved rollers made of hard gun-metal, 4 inches in length by $2\frac{1}{2}$ inches in diameter, mounted in bearings in a strong steel frame, *C*. The grooves are V-shaped, eighteen to the inch, and $\frac{1}{20}$ inch deep, with rather sharp, smooth edges. The rollers can be geared together in the ratio 2 : 3 by means of the solid brass gear wheels *D* and *E*. The wheel *E* is perforated with a number of holes, *H*, shown in the side-elevation, and when moved along its shaft, so that the fixed pin *P* enters one of these holes, it is thrown out of gear, and the roller is fixed. The distance between the rollers *A* and *B* is capable of adjustment, and is regulated by means of the set screws, *SS*. The shafts of the rollers are lubricated by four small Stauffer lubricators (not shown in the figure), which are screwed into holes in the frame *C*.

The machine is firmly bolted to the bench, as shown in the figure, and the tray *T*, which slides into the space underneath the rollers, receives the powdered rubber.

The rubber for analysis is cut with scissors or a knife into pieces about $\frac{1}{8}$ inch thick. These are passed through the rollers about three times with the wheels in gear, which crushes and cuts the rubber without grinding it much. The grinding process is completed by passing the broken-up rubber again through the rollers with the back roller fixed, and most samples are in this way reduced to powder. Any large and imperfectly ground pieces are picked out and again passed through the rollers. It sometimes happens with very soft, pure rubber that the particles stick together, but they can generally be easily separated again by the fingers. I have not had any experience in the analysis of raw rubber, but all samples of vulcanised rubber goods which I have analysed have been successfully prepared by this machine. Not more than ten minutes are required to prepare a sufficient quantity of the material for an ordinary analysis. As examples of rubbers prepared in this way I exhibit the following:—



Rubber-Grinding Machine.

Description of Article.	Mineral Matter (Ash).	Condition when Ground.
	Per Cent.	
Washer	52	Fine, non-adherent powder.
Sheeting, good quality	40	Somewhat adherent powder.
,, low quality	46	Fine, non-adherent powder.
Soft red tubing	11	Somewhat adherent powder.
Motor tyre inner tube	9	,, ,, ,,
Very elastic black tubing ...	1	Very adherent particles.

Even the last sample of pure vulcanised rubber has been reduced to a condition in which the reagents can attack it without difficulty.

I have had some experiments made to ascertain the effect of grinding on the figures obtained by analysis, the material used being a rubber washer composed of about 50 per cent. rubber and sulphur and 50 per cent. mineral matter (calcium carbonate and lead oxide).

	Acetone Extract, Less Free Sulphur.	Free Sulphur.	Alkali Extract.
	Per Cent.	Per Cent.	Per Cent.
Cut up with scissors into thin strips	2·69	1·51	1·31
Ground for 10 minutes	2·85	1·33	1·78
,, 15 ,, ...	3·06	1·69	1·56
,, 30 ,, ...	4·40	1·18	1·56

The prepared rubber was first extracted for eight hours in a Soxhlet extractor with boiling acetone, and the extracted rubber, after carefully evaporating off the whole of the adhering acetone, was boiled for four hours with 6 per cent. alcoholic soda solution. The percentage of saponified organic matter, freed from mineral matter, recovered from this solution is given in the column headed "Alkali Extract." Prolonged grinding, as would be expected, causes changes to take place which increase the percentage of matters extracted by acetone; but ten minutes' grinding has not appreciably altered the rubber. The grinding has not caused any chemical changes tending to increase the alkali extract, the higher figures obtained with the ground material being obviously due to the finer division of the sample and the more complete extraction. The "free sulphur" figures are peculiar, and I can offer no explanation of them. They were obtained by my assistant, Mr. W. H. Keys, who is a very careful worker, and I have no doubt they accurately represent the amount of sulphur extracted in each case by the acetone. The conclusion to be drawn from these experiments is that the amount of grinding needed to prepare a sample of rubber with the mill I have described does not materially alter the chemical composition of the rubber.

It may be of interest to place on record some further figures showing that eight hours' extraction with acetone is sufficient for practical purposes in the analysis of vulcanised rubber goods.

Description of Article.	Total Acetone Extract, including Free Sulphur.	
	Eight Hours' Extraction.	Sixteen Hours' Extraction.
	Per Cent.	Per Cent.
Rubber hose, canvas removed ...	7·92 7·97	8·12
Rubber washer	4·15 4·03 4·18	4·45
Gas tubing	2·97 2·93	3·02

Incidentally, the above figures show how closely concordant are the results of repeated analyses of the same material. Weber and others have laid stress upon the difficulty of obtaining an average sample of a rubber article. Weber goes so far as to say that the proportion of the various constituents is scarcely ever the same throughout the sample, and Henriques some years ago mentioned a case in which the percentage of silicious matter in a rubber sheet varied from 1·4 in one place to 28 in another. These observations do not accord with my experience. I have had occasion to analyse samples taken from successive consignments of many different kinds of rubber goods from various makers, and I have been surprised to find the uniformity in composition of the same kind of goods supplied by the same maker. I could bring much evidence in proof of this, but the following will suffice :—

In the Midland Railway specifications for certain kinds of rubber goods we ask for a test-piece to be supplied which is to be made from the same "mixing" as the goods and vulcanised under the same conditions. These test-pieces are required for mechanical tests. I almost invariably analyse them, and compare the composition with that of the goods they are supposed to represent. The number of instances in which the analysis of the test-piece is practically identical with that of a sample of the goods is so great as to prove, in my opinion, that the production of a uniform mixture presents no difficulty to the manufacturer, and that the analysis of a small piece of properly manufactured rubber may be taken as representative of the whole.

Discussion.

Mr. C. **Beadle** said that prolonged grinding caused the particles of some rubbers to mat together, and it might be that the diminution in the free sulphur was due to this cause.

Mr. A. C. CHAPMAN remarked that the process of extracting with acetone might in the case of certain rubbers be affected to an important extent by the tendency of the particles of rubber to clot together, the surface presented to the solvent being thus reduced. This could be overcome by spreading the finely divided rubber on a piece of wire gauze, rolling this up, and placing the whole in the Soxhlet apparatus.

Mr. BEVAN said that the sulphur might act on the bronze of the rollers and be driven off in a gaseous form by the heat produced during grinding. The matting together of the rubber particles might perhaps be prevented by grinding with some inert material.

Dr. STEVENS suggested that rollers of chilled steel might be better than bronze. If the rollers became hot, as he should have thought would be the case with one fixed and the other revolving, the heat might alter the composition of the rubber. The exposure of a large area to the action of the solvent during extraction was generally sufficiently attained if the rubber was reduced to a thin sheet having a broken surface, and, in fact, this was what was really obtained on grinding soft rubber, since the very fine particles adhered together again. In grinding waste rubber the most effective gearing for the rollers was found to be 1 to 4, 5, or even 6, so that possibly some such ratio might be found more effective than the use of one fixed roller.

Mr. ARCHBUTT, in reply, said that various gears had been tried, and 3 to 2 had been found to be the best when both rollers were in gear. With regard to the use of the fixed roller, it was found, accidentally, in the case of a sample which could not be reduced to powder by putting it through the machine with both rollers in gear, that the desired effect was obtained when one roller was fixed. With some samples the preparation was easier with one roller fixed all the time, whilst with others the best results were obtained by first using the rollers in differential gear. Chilled steel might answer equally well, and a hard aluminium or manganese aluminium bronze even better than gun-metal, for the rollers. Steel would be troublesome to keep clean in the laboratory atmosphere. The gun-metal rollers he had used showed no sign of tarnish or of any action of sulphur, though a very large number of samples had been passed through them. They do not get hot. He agreed as to the probable advantage of grinding with the addition of some inert material in the case of pure rubber, though he did not have to examine many such samples.

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THE DETECTION AND ESTIMATION OF BENZOIC ACID IN MILK AND CREAM.

By E. HINKS, B.Sc., F.I.C.

(*Read at the Meeting, November* 5, 1913.)

During the last few years several methods have been proposed for the detection of benzoic acid in milk and cream, notably by H. D. Richmond (Analyst, 1907, **32**, 151), C. Revis (*ibid.*, 1912, **37**, 346), and G. W. Monier-Williams (*Local Government Board Food Reports*, No. 10, 1909). These methods, though certain in their application, require for their performance a larger quantity of material than is often available for analysis, and do not deal with the quantitative aspect. The following methods are now proposed as being of equal, or enhanced, delicacy, with the use of smaller quantities of material, and as leading to quantitative results of a satisfactory degree of accuracy.

Qualitative.—Twenty-five c.c. of milk, or from 10 to 20 grms. of cream, are heated with an equal volume of concentrated hydrochloric acid, as in the Werner-Schmidt method for the estimation of fat, until the precipitated curd is completely dissolved. The cooled mixture is shaken with 25 c.c. of mixed ether and petroleum spirit (see below). The ethereal solution is separated, 1 drop of ammonia solution added, followed by 5 c.c. of water. The mixture is shaken, the aqueous layer separated, heated for a few minutes on the water-bath to expel excess of ammonia, and tested for benzoic acid with ferric chloride in the usual manner.

On the addition of the ammonia (before the addition of the water) a precipitation of ammonium benzoate takes place in the ethereal solution, if benzoic acid be present. The greater the proportion of petroleum spirit relatively to methylated ether, the more pronounced becomes the benzoate precipitate; but, on the other hand, the less marked is the final precipitate of ferric benzoate. Petroleum spirit alone gives a decreased delicacy as regards the ammonium precipitate, and failure as regards the iron precipitate; methylated ether alone fails (or nearly so) as regards the ammonium benzoate precipitate, but yields a good iron precipitate. A mixture of 2 vols. of petroleum spirit and 1 vol. of methylated ether gives good precipitates with both reagents, and is recommended for use. Of the two reactions, the ammonium benzoate precipitation, though less characteristic, is the more delicate. If 25 c.c. of milk be treated as above, 0·01 per cent. of benzoic acid may with certainty be detected, a similar order of delicacy being obtainable in the case of cream. A milk or cream free from benzoate often gives, on the addition of the ammonia, an opalescence, and gradually a slight precipitate, which is, however, readily distinguished from the precipitate yielded by benzoic acid. In doubtful cases one must proceed to the iron reaction.

If reliance be placed on the ammonium precipitate, it is advisable to use 0·880 ammonia solution.

Quantitative.—From 25 to 50 c.c. of milk, or from 10 to 20 grms. of cream, are heated with an equal volume of hydrochloric acid, as in the qualitative test. To avoid possible loss of benzoic acid this operation is conducted under a reflux condenser, although the loss without a condenser is very small. The cooled mixture is extracted three times with 20 c.c. of a mixture of equal volumes of ether and petroleum spirit; the mixed separated "ethereal" layers are rendered alkaline with ammonia solution, 10 c.c. of water added, and the mixture shaken; the aqueous layer is separated and the extraction repeated twice, keeping alkaline with ammonia if required. The mixed aqueous portions are made acid with hydrochloric acid and extracted three times with 20 c.c. of the mixed ether and spirit as before. The final mixed "ethereal" solutions are allowed to evaporate spontaneously and dried in a desiccator until constant in weight (twenty-four hours is sufficient as a rule for the air-dried residue). The benzoic acid is then driven off by heating at 100° C. (an hour or two), and the loss on heating gives the amount of benzoic acid present. Treated in this way, milk and cream free from benzoate give a "desiccated residue" of only a few mgrms., which should lose only a fraction of a mgrm. on heating at 100° C.

By performing the first part of the volatilisation of the benzoic acid on a hot plate, and covering the dish with a glass, much of the benzoic acid may be recovered and be proved to be benzoic acid in the usual manner, the volatilisation being then completed in the oven at 100° C.

The use of petroleum spirit alone for the extractions is inadmissible; methylated ether alone may be used. A mixture of equal volumes of the two is recommended.

The following table gives the results obtained by the above method with milks and creams containing known amounts of benzoic acid:—

Benzoic Acid Present.	Amount of Milk taken for Analysis.	Benzoic Acid Found.
Per Cent.	c.c.	Per Cent.
0·010	50	0·008
0·025	50	0·027
0·050	25	0·052
0·050*	25	0·050
0·100	25	0·100
0·200	25	0·191
0·100	20 grms. cream	0·098
0·250	10 ,,	0·245
0·500	10 ,,	0·495

The method gives a close approximation to accuracy in the second place of decimals.

The benzoic acid may also be determined by titration of the final residue, but the sublimation method is to be preferred.

The loss that is liable to occur in the various stages of two processes for the detection of benzoic acid referred to at the outset is seen in the following experiments:—

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* This milk was analysed five months after the addition of the benzoic acid.

1. To 50 c.c. of milk 0·025 grm. of benzoic acid dissolved in sodium carbonate solution was added. The curd was precipitated by acetic acid (Richmond, *loc. cit.*). The benzoic acid in this curd was (on digestion with hydrochloric acid) found to be 0·004 grm.

2. To 100 c.c. of milk 0·050 grm. of benzoic acid in sodium carbonate solution was added, and the milk treated according to Revis (*loc. cit.*). The benzoic acid was then determined by the hydrochloric acid process in the precipitates and final solution obtained. The calcium precipitate was found to contain 0·0068 grm. benzoic acid, the copper precipitate 0·0015 grm., and the final solution yielded 0·0413 grm., totalling 0·0496 grm.

The presence of boric acid does not interfere with the method described. As is to be expected, salicylic acid behaves in a manner similar to benzoic, though owing to the very great delicacy of the iron reaction with salicylic acid, smaller quantities of the latter acid may be detected. Salicylic acid appears to require a greater number of extractions by the solvents used in order to insure complete extraction.

Butter.—The method has been applied (in two experiments only) to butter. The butter may be treated direct as in the case of cream, or the aqueous portion separated, the fat shaken several times with hot sodium bicarbonate solution, and the mixed washings and aqueous portions digested with hydrochloric acid. The results of these two trials were:—

		Benzoic Acid Found.	
Benzoic Acid Present.	Butter taken for Analysis.	Direct Method.	Sodium Bicarbonate Method.
Per Cent.	Grms.	Per Cent.	Per Cent.
0·30	10	—	0·28
0·20	10	0·22	0·18

DISCUSSION.

Mr. C. REVIS said that this method seemed to be as expeditious as any he knew of, and apparently was free from a difficulty which beset most—namely, the solubility of the benzoic acid in the fat, which probably accounted for the low results often obtained in the case of butter. He should like to hear whether any other substance besides benzoic acid would be obtained by Mr. Hinks's method of treatment, as his experience was that a certain extract was always obtained in such cases, even in the absence of benzoic acid. Cinnamic acid, for instance, which was now being sold as a cream preservative, gave reactions very similar to those of benzoic acid.

Dr. E. K. RIDEAL said that in a recently devised method for the preparation of ammonium salts of aliphatic acids, dry ammonia gas was passed into the acids dissolved or suspended in a mixture of alcohol and petroleum spirit, the ammonium salts being precipitated in this semi-colloidal form; and it seemed likely that in Mr. Hinks's process small quantities of lactic acid, and possibly of other acids of the same series, might be formed. This would account for the small amount of residue left when milk was decomposed in the manner described in the paper.

Mr. Hinks said the residue left in the blank determination was doubtless due to a little lactic acid and fatty acid, but not more than 2 mgrms. should be obtained, which lost only a fraction of a mgrm. on heating. He had not tried whether cinnamic acid was extracted by this treatment, but he should imagine that it would behave similarly to benzoic acid.

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Calculation of the Quantity of Cocoanut Oil Present in a Butter from the Polenske Value of the Mixture. T. Sundberg. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1913, **26**, 422-424.)—The author finds, as the result of the analyses of numerous mixtures of cocoanut oil and butter fat, that the ordinary method of calculating the quantity of cocoanut oil in the mixture from the Polenske value yields too high results. For instance, a mixture containing 20 per cent. of cocoanut oil yielded a Reichert-Meissl value of 23·7, and a Polenske value of 4·4; according to Polenske's table, a Reichert-Meissl value of 23·7 corresponds with a Polenske value of 1·7, and the quantity of cocoanut oil in the mixture would be: $(4{\cdot}4 - 1{\cdot}7) \times 10$, that is, 27 per cent. The formula, however, really gives the amount of cocoanut oil expressed as a percentage of the butter fat present, and the quantity present in the mixture would be: $\frac{27 \times 100}{100 + 27}$, or 21·3 per cent. This method of calculation is trustworthy for mixtures containing up to 40 per cent. of cocoanut oil. For example, a mixture containing 41 per cent. gave 38 per cent., calculated by the new formula, whilst the old formula showed 62 per cent. of cocoanut oil to be present.

W. P. S.

Rapid and Accurate Method for Determining Fat in Ice Creams, Cereals, and Chocolate. E. P. Harding and G. Parkin. (*J. Ind. and Eng. Chem.*, 1913, **5**, 843-845.)—The paper is a continuation of one already published by the authors (Analyst, 1913, 147), where the method was employed on evaporated milk and milk powders. With the following modifications it can be used for ice cream:—The material is melted by warming to 50° C., and well mixed without churning; 5 grms. are weighed into the 100 c.c. extraction-flask, 5 c.c. of 25 per cent. acetic acid added, and the whole cautiously warmed to about 50° C. When the protein has dissolved, 12 c.c. of carbon tetrachloride are added, followed by 10 c.c. of alcohol and 25 c.c. of petroleum spirit; the rest of the procedure is the same as before. A complete determination can be made in an hour or an hour and a half. The percentage of fat is slightly higher, and the fat itself is purer than with the Röse-Gottlieb or Babcock methods. When applied to cereals, fertilisers, and chocolate, hydrochloric acid of sp. gr. 1·12 must be substituted for the acetic acid, since the latter acid will not liberate the oil successfully. With chocolates it is necessary to centrifuge for two minutes to obtain the first separation, and the resulting fat from this last-named material, though slightly smaller in amount than that obtainable by the Bigelow and Albrech method (*A.C.A.C. Bull.*, No. 137, pp. 102-103) used for comparison, was free from the dirty appearance and admixed fibre associated with this method. More than three successive washings with ether are unnecessary.

H. F. E. H.

Essential Oils from Various Countries. (*Bull. Imp. Inst.*, 1913, **11**, 428, 439.)—Samples of essential oils from Cyprus, Hong-Kong, West Indies, and Zanzibar, gave the following analytical values:—

Oil.	Specific Gravity at 15°/15° C.	Optical Rotation, 100 mm. at 20° C.	Saponification Value.		Solubility in Alcohol.	
			Oil.	Acetylated Oil.		
Juniper oil, from *Juniperus Phœnicia*	0·8684	+ 3° 4′	9·8	16·8	Opalescent solution in 9 vols. of 90% alcohol	—
Sage oil, from *Salvia Cypria*	0·9263	− 6° 31′	13·9	38·9	In 1 or more vols. of 80% alcohol	Cineol, 75%
Laurel-leaf oil, from *Laurus nobilis* (three samples)	0·9224 to 0·940	− 4° 45′ to − 11° 32′	—	—	In 1 to 1·7 vols. of 80% alcohol	—
Mint oil, from *Mentha sylvestris*	0·9687	+ 31° 58′	24·9	175·5	In 2·5 vols. of 70% alcohol	—
Myrtle oil, from *Myrtus communis* (two samples)	0·9166 0·9302	+ 8° 14′ + 8°	25·1 25·2	61·5 —	In 5 or more vols. of 70% alcohol	—
Peppermint oil (Hong-Kong)	0·9235 0·9359	− 44° 8′ − 41° 5′	— —		In 2·5 vols. of 70% alcohol	Free menthol, 43·3% and 47·49%; menthol as esters, 14·35% and 16·35%
"Ngai Yau" oil, from *Artemisia vulgaris*	0·9390	—	103·3	205·87	In 1 vol. of 90% alcohol	—
"Petit Grain" oil, Jamaica	0·8884	− 6° 45′	—		In 1 vol.	Esters as linalyl acetate, 30·6%
Pimento-leaf oil ...	1·0260	− 5° 30′	—		In 1·6 vol. of 70% alcohol	Phenols, chiefly eugenol, 68·6%
Clove-leaf oil, Zanzibar	1·0652	—	—		In 1·1 vol. of 70% alcohol	Eugenol, 85·7%

C. A. M.

Microscopical Detection of Embelic Acid. G. Heyl and P. Kneip. (*Apoth. Zeit.*, 1913, **28**, 699; through *Chem. Zentralbl.*, 1913, II., 1342.)—Embelic acid, the active constituent of the fruit (employed as an anthelmintic) of the Indian plant *Embelia ribes Burm.* (Nat. Ord., *Myrsinaceæ*), may be isolated readily as the hydroxyquinone derivative by microsublimation, and identified microchemically. The microsublimates obtained by Tunmann's method were partly crystalline bundles and partly tabular crystalline structures, surrounded by a yellowish mass (illustrated in the original). They are insoluble in water, but dissolve with a reddish-violet coloration in a mixture of 1 drop of 15 per cent. sodium hydroxide with 10 c.c. of water; treatment with 15 per cent. sodium hydroxide causes the precipitation of small shining violet plates, after a time, mixed with needles of the same colour. The addition of hydrochloric acid causes the separation of a flocculent precipitate of embelic acid. Carbonates of the alkali metals produce the same effect as the hydroxides. With dilute ammonia, a light red solution is obtained; on evaporation, small crystalline bundles gradually separate. The dilute alkaline solution of the sublimates gives an olive-brown precipitate with dilute copper sulphate solution, with dilute barium chloride a grey-brown, with dilute nickel sulphate a green-brown, and with magnesium sulphate a flocculent brown precipitate. The alcoholic solution of the sublimate gives with ferric chloride a red-brown, with copper nitrate a dirty green, with lead acetate a dark green, and with zinc chloride a violet precipitate. Mercuric chloride and silver nitrate cause no precipitation. The violet coloration produced by warming embelic acid itself with sulphuric acid only occurs indistinctly with the sublimate.

O. E. M.

Testing Ether. G. Frerichs. (*Apoth. Zeit.*, 1913, **28**, 628-630; through *Chem. Zentralbl.*, 1913, II., 1254-1255.)—In testing ether it may be required to ascertain whether it has been prepared from pure alcohol or methylated spirit. In Germany the denaturing agent is a mixture of crude wood spirit and pyridine bases, the latter remaining in combination with the sulphuric acid when the ether is made. The methyl alcohol and acetone of the wood spirit appear in the product as methyl-ethyl ether and unaltered acetone. The former produces a considerable depression of the boiling-point. Perfectly pure ether boils under the normal pressure at 34·2° to 34·3° C. The presence of 1 per cent. of alcohol raises the boiling-point 0·2° C., so that the boiling-point of 35° C., prescribed by the German codex, would permit of the presence of 4 per cent. of alcohol. Such an ether would, however, show a density of 0·723 instead of 0·720. The official method for determining the boiling-point is not sufficiently sharp; it is better to distil off the first 50 c.c. from 250 c.c. of ether and use this fraction for the determination of the boiling-point. A still more accurate method is to determine the boiling-point under a reflux condenser. A special form of boiling-flask is employed having two necks, in one of which the thermometer is inserted, whilst the other is connected with the reflux condenser, the two necks being joined together near the top by a branch tube. For the detection of acetone in ether, 100 c.c. of the ether are shaken vigorously with 10 c.c. of water, the two layers are allowed to separate, and the water is collected in two test-tubes. To one portion the sodium nitroprusside test is applied, to the other the iodoform test.

J. F. B.

Non-Nitrogenous Extractive Matters in Fodder and Foodstuffs. J. König. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1913, **26**, 273-281.)—The group of so-called non-nitrogenous extractive matters is of somewhat indefinite composition, since it represents only the difference between 100 and the sum of the percentages of moisture, proteins, fats, crude fibre, and mineral matters, and consequently varies according to the effectiveness of the methods by which all these are determined. To a certain extent the term "extractive" matter is a misnomer, because a considerable portion of this group is not extracted directly, but is rendered soluble by hydrolysis more or less profound. Some portions of the group—*e.g.* sugars, dextrins, starch, inulin, various organic acids—are sufficiently definite and well characterised; others —*e.g.* pectins, tannins, bitter principles, colouring matters—are not so well known, but still have a generally defined significance. On the other hand, the pentosans, hemicelluloses, and a portion of the lignin, occur in various degrees of condensation, and may appear partly in the "extractives" and partly in the crude fibre, according to the methods employed. The pentosans are easily and completely estimated by the distillation method, and it is therefore desirable to adopt means whereby they are totally excluded from the crude fibre value, in order to avoid reckoning them twice over. This object is almost completely effected by the author's glycerol and sulphuric acid digestion method, which nevertheless gives results practically identical with the Weende method. The reason is that König's crude fibre, though practically free from pentosans, which are readily digestible by animals, includes more of the lignin, which is almost undigestible, than the Weende crude fibre. The cell walls of plants consist of bodies belonging to four groups; pentosans, hexosans, lignins, and cutins. So far as the first three of these are concerned they exist in different degrees of solubility (condensation, or resistance to hydrolysis). These degrees must necessarily overlap, and a sharp separation must be difficult, if not impossible. Portions of all three groups are soluble on digestion with water alone under a pressure of 2 to 3 atmospheres, also on digestion with enzymes such as diastase or pepsin-hydrochloric acid. The members of this group correspond with starch, and are termed by the author protopentosans, protocelluloses (hexosans), and protolignins. The bodies of the second group are rendered soluble by digestion with dilute (2 to 3 per cent.) acids under pressure, or by boiling at 135° to 137° C. These are called hemipentosans, hemicelluloses (hexosans), and hemilignins. König's glycerol-sulphuric acid method affords an approximate separation of the first two groups from the third or ortho group, which constitutes, together with the cutin, the so-called "crude fibre." The orthopentosans occur only in very small proportions; the orthohexosans, so far, have not been observed to include any anhydrides of mannose and galactose, and may therefore be said to consist substantially of orthocellulose, the "true" cellulose. The ortholignins are oxidisable bodies, and may be separated from the orthocellulose by suitable treatment with ammoniacal hydrogen peroxide; these lignins may be split up into two groups by treatment of the crude fibre with 72 per cent. sulphuric acid. This acid dissolves the orthopentosans, the orthocellulose, and a portion (colourless) of the ortholignins, leaving a residue of dark coloured ortholignins insoluble. The cutin is susceptible neither to hydrolysis nor oxidation under the conditions specified, nor is it soluble in the solvents of cellulose. In view of the fact that a sharp separation of the groups of various degrees of resistance in impossible, it is highly desirable that an international standard method for their approximate classification be adopted, so that the results may at least be comparable. J. F. B.

Hop Analysis. O. Neumann. (*Chem. Zeit.*, 1913, **37**, 1317-1318.)—The author cannot accept the conclusions of Power, Tutin, and Rogerson (ANALYST, 1913, 459), that the petroleum spirit extract of hops does not contain the bitter principles (? resins), but consists mainly of fatty acids. Such conclusions are contrary to established experience, which leaves no doubt as to the utility of the estimation of bitter principles in hop analysis. The tests for bitter principles and estimations of lupulin always give concordant results. Hops poor in lupulin never show a high percentage of bitter principles, and those rich in lupulin are never poor in bitters. Seibriger has found that carbon tetrachloride is equivalent both quantitatively and qualitatively to petroleum spirit as a solvent, and possesses the advantage of uniformity of composition; moreover, the time of extraction may be reduced from seven hours to three hours. The hops are chopped up in a mincing-machine with sharp knives, and 10 grms. are shaken in the cold with the solvent, whether petroleum spirit or carbon tetrachloride. For this purpose an apparatus such as that designed by Wagner for the estimation of phosphoric acid in fertilisers is most suitable. The speed of rotation is regulated at 15 revolutions per minute. For the titration with alcoholic potassium hydroxide, 100 c.c. of the filtrate are taken. In order to avoid the separation of layers 80 c.c. of neutral 96 per cent. alcohol are added before the titration. The extraction of the bitter principles can be effected with cold petroleum spirit by shaking for three or four hours. With carbon tetrachloride one or two hours' shaking is sufficient. The results with petroleum spirit are slightly lower than with carbon tetrachloride, but the divergences do not exceed the permissible limits. Extraction in the cold constitutes a notable simplification in hop analysis. J. F. B.

Direct Iron Estimation in *Ferri et Quininae Citras* and *Extractum Ferri Pomatum*. F. Lehmann. (*Apoth. Zeit.*, 1913, **28**, 708; through *Chem. Zentralbl.*, 1913, II., 1342.)—The author's method (*Apoth. Zeit.*, 1911, **26**, 125; *Chem. Zentralbl.*, 1911, I., 845) for the destruction of the lactic acid radical in *ferrum lacticum* by means of hydrogen peroxide and sulphuric acid may be applied to the destruction of the organic portion of iron and quinine citrate, and in iron lactate extract. (1) *Ferri et Quininæ Citras.*—0·4 grm. of the sample is dissolved in 10 c.c. of water, boiled, 30 c.c. of 4 per cent. hydrogen peroxide added, and the liquid shaken and allowed to stand with frequent agitation until gas evolution ceases; it is then boiled with 5 c.c. of concentrated sulphuric acid, and, after excess of hydrogen peroxide has been removed by means of potassium permanganate added drop by drop, allowed to stand for one hour with 2 grms. of potassium iodide in a closed flask. The iodine is then titrated back with $\frac{N}{10}$ thiosulphate and starch; 1 c.c. $\frac{N}{10}$ thiosulphate corresponds to 0·0056 grm. iron. (2) *Extractum Ferri Pomatum.*—The method is the same, 1 grm. of extract being used. O. E. M.

Detection of Potassium Dichromate in Milk. B. Grewing. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1913, **26**, 287.)—Ten c.c. of the milk are mixed with 4 c.c. of a 3 per cent. aniline solution, and the mixture is poured on the surface of sulphuric acid contained in a test-tube. A blue zone develops at the surface of contact of the two liquids if dichromate is present; a violet zone below the blue zone will also be noticed. When the milk contains from 0·05 to 0·1 grm. of dichromate per litre, a green zone appears first, and changes gradually to blue; with 0·01 grm. of the salt per litre, the coloration obtained is red-violet, and develops after the lapse of about six minutes. The presence of nitrates, formaldehyde, and hydrogen peroxide, does not interfere with the reaction. W. P. S.

Detection of Nitric Acid (Nitrates) in Milk. R. Barth. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1913, **26**, 339-341.)—The formaldehyde-sulphuric acid test is recommended (*cf.* ANALYST, 1902, **27**, 357) as being capable of detecting as little as 0·5 mgrm. of nitric acid per litre of milk. In order to prevent the formation of a brown ring at the point of contact of the acid with the milk, sulphuric acid having a sp. gr. of 1·71 should be used. The water employed in reducing concentrated acid to this strength must be free from nitric acid, and, as ordinary distilled water frequently contains traces of nitric acid, it is advisable to re-distil the water after the addition of a small quantity of sodium hydroxide. In carrying out the test, 10 c.c. of the milk are mixed with 5 drops of formaldehyde solution (prepared by adding 10 drops of commercial formalin to 250 c.c. of water), and the mixture is poured carefully on the surface of 5 c.c. of the sulphuric acid contained in a test-tube. With 0·5 mgrm. of nitric acid per litre, the blue ring appears after the lapse of about five minutes; with 5 mgrms. of nitric acid per litre the blue ring develops almost immediately.

W. P. S.

Estimation of Phosphorus in Manures and Feeding-Stuffs. C. Duserre and P. Chavan. (*Mitt. Lebensmitteluntersuch. Hyg.*, 1913, **4**, 261; through *Chem. Zentralbl.*, 1913, II., 1333.)—For the estimation of phosphoric acid the modification of Pemberton's method recommended by Molinari, consisting in the addition of Petermann solution to the liquid to be precipitated, gave good results with superphosphates, but the authors occasionally found high values when working in sulphuric acid solution with "Thomas" phosphate or bone meal. By adding ammonium sulphate instead of nitrate to the molybdenum solution, they obtained accurate "results." The solution was prepared by dissolving 25 grms. ammonium sulphate in 400 c.c. nitric acid of density 1·2, and 75 grms. ammonium molybdate in 500 c.c. of warm water, and mixing when cool. Tables are given showing the distribution of phosphorus-containing compounds in hay from a meadow not manured, and from the same meadow manured with superphosphate. The phosphorus present as lecithin was determined by extraction of the hay for one hour with boiling ether, repeated five times, and treatment of the residue from the ethereal extract with a mixture of nitric and sulphuric acid; the hay exhausted with ether was then extracted for one hour with boiling absolute alcohol, the extract evaporated down, and the residue treated as before. To determine the inorganic phosphorus, the hay thus treated was extracted with 1 per cent. cold hydrochloric acid. The phosphorus present as nuclein was determined by treatment of the extracted hay with nitrosulphuric acid. For the estimation of phosphorus in organic compounds the following is recommended as leading to the least loss of phosphorus: 5 to 10 grms. of the substance are heated in a 300-400 c.c. Kjeldahl flask with 15 c.c. of sulpuric acid and 15 c.c. of nitric acid; nitric acid is added drop by drop when the nitrous fumes have disappeared. Water is added to the clear liquid, and the whole boiled; after cooling, precipitation with the molybdate solution is carried out. O. E. M.

Estimation of Higher Alcohols in Spirits. R. Ofner. (*Chem. Zeit.*, 1913, **37**, 1239.)—The author has used the Komarowsky reaction (*cf.* ANALYST, 1910, **35**, 403) for the estimation of higher alcohols in spirits, and finds that the method is sufficiently accurate for most purposes. For the removal of aldehydes, the distilled spirit should be treated with *m*-phenylenediamine hydrochloride and re-distilled; a smaller quantity of salicylic aldehyde than that mentioned by Fellenberg (*loc. cit.*) should be used, and the spirit under examination and the comparison solutions should contain 50 per cent. of alcohol or be brought to this strength by suitable dilution. The following method also yields trustworthy results:—Ten c.c. of the distillate, after removal of aldehydes by means of *m*-phenylenediamine hydrochloride, are treated with 1 c.c. of a furfuraldehyde solution (1 grm. of furfuraldehyde in 1 litre of 50 per cent. alcohol), 10 c.c. of concentrated sulphuric acid are added, and the mixture is shaken and heated for twenty seconds over a flame. After cooling, the coloration is compared with those obtained under similar conditions with known quantities of amyl alcohol dissolved in 50 per cent. alcohol. In the case of cognac, isobutyl alcohol should be used in place of amyl alcohol in preparing the comparison solutions.

W. P. S.

Turpentine Sold as a Drug. (*Bull.* 253, *Inland Rev. Dept.*, Ottawa; through *J. Soc. Chem. Ind.*, 1913, **32**, 1028.)—The standards sanctioned for turpentine for medical purposes by the Privy Council in 1912 are—(1) It shall be entirely free from mineral oil. (2) Unless sold as wood turpentine, it shall absorb not less than 340 times its weight of iodine (Hübl solution and method); if sold as wood turpentine, it shall absorb not less than 240 times its weight of iodine by the same method. (3) The undissolved (unpolymerised) residue on treatment of 10 c.c. with 40 c.c. of a sulphuric acid containing 20 per cent. of fuming acid shall not exceed 10 per cent. by volume of the sample. (4) The refractive index of this residue shall be not less than 1·495 at 20° C. (5) The refractive index of the sample at 20° C. shall lie between 1·468 and 1·473. (6) The sp. gr. of the sample at 20° C. shall not be less than 0·86. (7) The initial boiling-point shall not be lower than 150° C. under ordinary atmospheric pressure. (8) At least 75 per cent. by volume shall distil below 160° C. (9) The residue on evaporation over a steam-bath shall not exceed 2 per cent. Forty-two samples out of 158 collected were found to contain petroleum, and ten other samples did not comply with the above requirements.

Detection of Adulteration of Cider Vinegar with Distilled Vinegar. S. L. Crawford. (*J. Ind. and Eng. Chem.*, 1913, **5**, 845-847.)—The volatile constituent with a reducing action on Fehling's solution, which is found in all pure cider vinegars, is absent from distilled vinegar and pyroligneous acid. The fact that it is also absent from fully fermented dry refined cider, six months old (acidity 0·36 per cent.), and from completely fermented first-pressing juice six months old but not refined (acidity 0·75 per cent.), goes to show that this volatile reducing substance is formed during the acetification of the apple juice. The proposed method is to dilute 50 c.c. of the sample to 250 c.c., and distil off 200 c.c. into a 250 c.c. flask, neutralise and make up to volume. The reducing substances are estimated in 50 c.c. of the distillate by Munson and Walker's method (*Bureau of Chem. Bull.*, 107). When reduced to a basis of 4 per cent. acidity (grms. per 100 c.c. of acetic acid), the distillates from nine samples of genuine cider vinegars are shown to contain from 0·111 to 0·149 per cent. of reducing substances calculated as "invert sugar." Taken in conjunction with the glycerol values and other constants, indications should be thus obtained as to the adulteration of cider vinegar with distilled vinegar or pyroligneous acid. If the latter adulterant has been employed, the vinegar will contain more formic acid than the amount usually found in cider vinegar (0·007 grm. per 100 c.c.), while if distilled vinegar is present the formic acid will be less than this amount to an extent dependent on the amount of cider vinegar present.

H. F. E. H.

So-called Non-Alcoholic Wines of Commerce. W. I. Baragiola and W. Boller. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1913, **26**, 369-408.)—The authors have submitted two samples of commercial "non-alcoholic wine" (pasteurised and clarified grape-juice) to an exhaustive analytical and chemico-physical investigation, both in their natural state and after fermentation with yeast. The analytical results have been used for calculating "balance sheets" of extract, acids, and mineral matters, for which purpose the methods of calculation adopted in the case of normal wines was found to be valid. The concentration of hydrogen ions or degree of acidity was estimated by the sucrose inversion method. One of these grape-juices in its original condition, and both of them after fermentation, showed, as compared with natural wines, an abnormally high concentration of hydrogen ions in relation to the proportion of titratable acid present. This high degree of dissociation of the acids is reflected in the flavour of the "non-alcoholic wines," which, in spite of the presence of sugar, always shows a tendency to be sharp. The sharpness would appear to depend upon a poverty in ammonium salts or organic bases, which have a powerful influence in depressing the ionisation of the acids without reducing their quantity. An investigation of a large number of samples of "non-alcoholic wines," with regard to the nitrogenous matters in various types of combination, might decide whether poverty in nitrogen is a direct consequence of the process of manufacture. It is suggested that a better conservation of the nitrogenous components might be aimed at in the process of pasteurisation and clarification, and might result in an improved and milder flavour. The "wines" before and after fermentation were also investigated according to the conductivity method of Dutoit and Duboux by titration with barium hydroxide, and the relationships of the conductivity curves are discussed in connection with the chemical composition; the influence of the sugar in the unfermented "wines" reduces the sharpness of the curves. In the determination of the ash it is shown, by mathematical treatment, that the errors are of the same order as in the analysis of ordinary wines. The chemico-physical balancing of the acids and bases was calculated by two distinct methods—Quartároli's, and Dutoit and Duboux's (ANALYST, 1910, **35**, 254)—with concordant results, and a representation of the condition of combination of the various organic and inorganic acids has thereby been obtained. The presence of very considerable quantities of tartaric acid in the free state is thus indicated in the grape-juices, whereas, according to the older methods of wine analysis, such juices would contain little or no free tartaric acid. J. F. B.

Wine Statistics for the Years 1911-1912. (*Arbeit. Kaiserl. Gesundheitsamte*, 1913, I., 555.)—The present report gives the proceedings of the Official Commission on Wine Statistics, together with details of analyses of 1,912 samples of wine and 6,087 samples of must obtained from the different wine-growing districts. Details are also given regarding the localities of the vineyards, the soil and manures used, kind of vines grown, insect pests and remedies for the same, vintage, climatic notes, etc. Particular attention was paid to the amount of sulphurous acid in the wines; in most cases the total quantity of the acid was less than 200 mgrms. per litre, but in certain samples the quantity exceeded 300 mgrms. per litre. Deniges' test for citric acid was found to be trustworthy, but wines containing a large quantity of tannin must be treated with animal charcoal to remove this constituent before the test is applied. Further experience of the use of nicotine as an insecticide (*cf.* ANALYST, 1912, **37**, 96) has been of a satisfactory nature, and by repeated sprayings at proper intervals it has been possible to eliminate injurious insects from entire districts. W. P. S.

Accurate Estimation of the Dry Extract of Wines and Fermented Liquors. P. Malvezin. (*Bull. Soc. Chim.*, 1913, **13**, 943-947.)—None of the methods in use give constant results owing to the effect of the glycerol, which may or may not be partially removed. To obviate this error the amount of extract free from glycerol is estimated as follows:—Fifty c.c. of the wine are taken, and half of it is heated in a flask connected with a pump, and with a second flask which can be put out of communication by a stopcock. The wine is heated under reduced pressure at 75° to 80° C., until the residue is pasty, after which the second portion is introduced and the operation repeated. A current of steam is now introduced for an hour from the communicating flask. All the glycerol is thus expelled, and the residue is then dried and weighed. C. A. M.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Micro-Chemical Method for the Estimation of α- and β-Amino Acids and Certain Derivatives in Proteolysed Substances, Blood, and Urine. P. A. Kober and K. Sugiura. (*J. Amer. Chem. Soc.*, 1913, **35**, 1546-1584.)—The principle of the method is the property of α- and β-amino acids, peptides and peptones to dissolve cupric hydroxide quantitatively in the cold to form a metallic complex in a neutral or slightly alkaline solution ($C_H = 10^{-7 \cdot 07}$ to $10^{-8 \cdot 8}$). The reaction is extremely rapid and sensitive, and will give good results with 0·005 per cent. solutions of these substances, failure to obtain such a complex being proof of the absence of α- and β-amino-acids and their polypeptides. The extent to which the copper can be precipitated from the complexes of nineteen amino-acids, twenty-seven di-peptides, eighteen tri-peptides, and four tetra-peptides is given when in equilibrium with $\frac{N}{0 \cdot 06}$ barium hydroxide, and it is shown that the amount of copper dissolved by the free amino-acid can be distinguished from that dissolved by the peptides and peptones. Interfering substances are not numerous, the first class being dicarboxylic acids, and the second reducing substances such as the sugars and uric acid. The sugars can all be separated by ammoniacal lead acetate (5 c.c. strong ammonia to 25 c.c. 10 per cent. lead acetate), which does not throw down any amino-acids or peptides in very dilute solutions (1 in 25,000). An insoluble cuprous compound of uric acid is precipitated, when 2 c.c. of a 5 per cent. solution of cupric chloride and 0·5 c.c. saturated magnesium sulphate are added to such a body as urine. The method is shown to be applicable to proteolytic cleavage products, blood, and urine, so far as the accurate determination of amino-acids, peptides, and peptones in these materials is concerned, and may therefore be called a "clinical" method. Two very insoluble copper complexes, *n*-amino-caproic acid and phenylglycine copper, are described which may be found useful in analytical work with copper. By means of these two amino-acids the copper of all the other complexes, including those of the amino-acids and all the polypeptides thus far studied, with the exception of histidine, can be thrown down quantitatively. Therefore by means of these reagents histidine in very small quantities can be accurately and quickly determined. For following proteolysis the method is best used in conjunction with the nephelometric one of Kober (*J. Amer. Chem. Soc.*, 1913, **35**, 290; see p. 571). With this instrument can be estimated the amount of undigested protein at any time, while the total amino-acid nitrogen obtained by the copper method will indicate the amount of hydrolysis at any time. The copper complexes on treatment with barium hydroxide will give the amount of the free amino-acids—the final products of hydrolysis; all this can be done on 0·005 grm. of protein. For fuller details of the technique involved and of the solutions required the original paper should be consulted.
H. F. E. H.

Amino-Acids in Blood. A. Constontino. (*Biochem. Zeitsch.*, 1913, **55**, 419-424.)—The author recommends removal of proteins by means of an acid mercuric chloride solution before determining amino-acid nitrogen by Sorensen's formaldehyde method. Blood or serum (100 c.c.) is mixed with 500 c.c. of a 2 per cent. mercuric chloride solution containing 0·8 per cent. hydrochloric acid, vigorously shaken, and after standing with intermittent shaking for one hour is centrifuged, and filtered through a dry filter. A known volume of the filtrate is treated with hydrogen sulphide to remove mercury, the excess of hydrogen sulphide expelled by means of an air current, and the liquid, after being exactly neutralised, concentrated on the water-bath, or preferably at 50° C. under reduced pressure, and then distilled with magnesium oxide under reduced pressure at 45° C. to expel ammonia. E. W.

Detection of Blood. B. Bardach. (*Chem. Zeit.*, 1913, **37**, 1190-1191.)—The guaiacum-perborate test described previously by the author and Silberstein (Analyst, 1910, **35**, 439) is rendered more sensitive if the blood solution be treated previously with dilute acetic acid; the addition of pyridine, as recommended by Leers (*cf.* Michel, Analyst, 1912, **37**, 506), is also advantageous. Five c.c. of the blood solution (urine) are treated with 3 drops of 30 per cent. acetic acid, allowed to stand for about two minutes, and 2 drops of a saturated alcoholic guaiacum resin solution and 0·5 grm. of powdered sodium perborate are added. Three c.c. of 80 per cent. acetic acid are then added, the mixture is shaken, and 3 c.c. of alcohol are allowed to flow on to the surface of the solution, followed by 2 drops of pyridine. In the presence of blood, a blue zone appears within a few minutes, and then fades gradually. Nitrites yield a somewhat similar reaction, but in this case the lower layer of liquid is coloured blue, and the colour fades very rapidly, whilst with blood the coloration appears both at the junction of the liquids and in the alcoholic layer. Alkaline urines also yield a blue coloration with the test, but the colour develops immediately on the addition of the guaiacum tincture, perborate, and alcohol. When a positive reaction is obtained the presence of blood should be confirmed spectroscopically. The test is less sensitive in the case of urine than with aqueous blood solutions, but 0·017 c.c. of blood in 1 litre of urine gives a distinct reaction. W. P. S.

Cyanogenesis under Digestive Conditions. S. J. M. Auld. (*J. Agric. Sci.*, 1913, **5**, 409-417.)—Important foodstuffs which produce hydrocyanic acid in considerable quantity on maceration with water are linseed and linseed cake, improperly made linseed gruel, Java beans, and other varieties of *Phaseolus lunatus* and immature great millet (*Sorghum vulgare*). The generally innocuous character of linseed cake was explained by Henry and Auld (Analyst, 1908, **33**, 280) and Dunstan as due to the destruction by heat of the enzyme present during the hot process of expressing the oil. This is, however, not the case, as most linseed cakes do in fact liberate HCN in quantities varying from 0·001 to 0·052 per cent. on maceration with water. At blood heat such cakes will liberate *in vitro* half the available acid in fifteen minutes, and sheep fed with cake containing 0·052 per cent. of available HCN in quantities up to 4 pounds per day suffered no ill effects, although exposed to far more than the lethal dose. Investigation showed that under digestive conditions cyanogenesis is likely to be inhibited by acids and alkalis, digestive juices, cellulose (especially), glucose, molasses, salt, and many other feeding-stuff constituents and adjuncts. Owing to the time the food remains in the digestive tract (one to two hours) before coming to the true stomach or the acid-secreting portion of the stomach, normal inhibition of cyanogenesis is chiefly caused by the alkaline character (about $\frac{N}{10}$) of the saliva, and this explains the non-toxic character of linseed cake. Small amounts of hydrocyanic acid were found in the rumen of a sheep which was killed shortly after feeding on linseed cake, and it may be taken that cyanogenetic feeding-stuffs are most likely to be poisonous when fed with acid-containing or acid-producing foodstuffs, or where the HCN is already present, as in improperly made linseed gruel. Reasons are given for believing that the small quantities of HCN normally produced from cyanogenetic feeding-stuffs may possibly have a beneficial action, from their well-known tonic properties. H. F. E. H.

Estimation of *d*-Lactic Acid in Body Fluids and Organs. J. Yoshikawa.

(*Zeitsch. Physiol. Chem.*, 1913, **87**, 382-418.)—The *d*-lactic acid is estimated by measuring the rotation of a solution of its lithium salt. Tables and curves are given showing relationship between lactic acid content and rotation from which the following formulæ are derived:

$$a = -0{\cdot}286p + 0{\cdot}00342p^2 \ldots$$

$$p = \frac{0{\cdot}286 - \sqrt{0{\cdot}081796 + 0{\cdot}01368a}}{0{\cdot}00684}, \qquad a = -0{\cdot}2864c + 0{\cdot}00405c^2 \ldots$$

$$c = 0{\cdot}2864 - \frac{\sqrt{0{\cdot}082025 + 0{\cdot}0162a}}{0{\cdot}0081}.$$

p = weight per cent. of *d*-lithium lactate; d = density of solution; $c = p \times d$; a = angle of rotation.

The presence of inactive lactic acid or acetic acid do not affect the rotation. Urine was found to be free from *d*-lactic acid: the efficiency of the method was tested with urine to which a known quantity of acid had been added. By extracting with ether, after acidifying with phosphoric acid, 98·35 – 100·65 per cent. of the added acid was found. For the extraction of *d*-lactic acid from muscle, the author recommends the method of J. Mondschein (ANALYST, 1912, **37**, 403). The material is extracted with boiling water, and the extract and coagulum treated separately. The extract is concentrated to a thin syrup, and extracted with alcohol. After distilling off alcohol, the residue is acidified with phosphoric acid and extracted with ether, the ether extract distilled, and the residue dissolved in water and converted into lithium salt by treatment with lithium carbonate. The coagulum is dissolved by warming with sodium hydroxide, and the resulting solution freed from proteins by saturating the boiling liquid with salt and acidifying with sulphuric acid. The protein-free liquid is then treated in the same manner as the aqueous extract.

E. W.

Colorimetric Method for the Determination of Hydrocyanic Acid in Plants, with special reference to Kaffir Corn. C. K. Francis and W. B. Connell.

(*J. Amer. Chem. Soc.*, 1913, **35**, 1624-1628.)—The ferro-ferricyanide method gives variable results, and difficulty is experienced in keeping the ferrous sulphate solutions free from ferric compounds. The ammonia-alum (Allen's "Organic Analysis," 3rd. ed., vol. iii., p. 92) and the lead-acetate methods (*ibid.*) are only applicable when hydrocyanic acid is present in relatively large amounts (0·1 per cent. and over). The thiocyanate method, modified as follows, was finally adopted: Fifty grms. of the finely chopped material are triturated with 100 c.c. of water, and then washed into a litre distilling flask with 100 c.c. of water. Fifty c.c. of concentrated sulphuric acid are then added, and about 150 c.c. are distilled into 50 c.c. of a 4 per cent. solution of potassium hydroxide. The end of the condenser must be beneath the potash solution, or loss of hydrocyanic acid results. The distillate is made up to 250 c.c., and to 50 c.c. portions of this are added 1 c.c. of yellow ammonium sulphide, the whole being then taken to dryness on the water-bath. The residue is taken up with 10 or 15 c.c. of hot water and barely acidified with dilute hydrochloric acid. Free sulphur is removed by filtration, and 0·5 c.c. of dilute hydrochloric acid is added before boiling the solution for five minutes, after which sulphur is again filtered off, the boiling and filtering being repeated, if necessary, until the solution is absolutely clear. The solution is then nearly made up to the mark in a 50 c.c. Nessler tube, and 15 drops of a 5 per cent. ferric chloride solution are added, when a bright cherry-red coloration indicates the presence of cyanide in the sample. If too much acid is present the colour will be lemon yellow, and if alkaline, iron will be precipitated. The standard solution of potassium thiocyanate is made to contain 15 grms. of this salt in a litre and standardised gravimetrically with silver until 1 c.c. = 14·92 mgrms. KSCN = 10 mgrms. of KCN. Fifty c.c. of this solution are diluted for use in the Nessler tubes, 1 c.c. of the diluted salt being equivalent to a mgrm. of KCN. There is a tendency towards obtaining high results when using this method, but it compares favourably with the silver nitrate gravimetric method, and is very satisfactory for determinations of KCN in amounts under 0·5 per cent. Chlorides, bromides, and iodides do not interfere. Some results found by using the method on Kaffir corn plants are as follows:—

	HCN per cent.
Leaves. Plant stunted and frost-bitten	0·010
Stalk and leaves. Plant in good condition	0·0007
Entire plant without roots. Taken from field in which stock was poisoned. Plants much stunted and almost dry from lack of water. ...	0·0124

H. F. E. H.

Solubility of Milk Proteins in the Constituents of the Serum. L. Lindet. (*Bull. Soc. Chim.*, 1913, **13**, 929-935.)—Previous investigations have shown that the albumin of milk can only be distinguished from the casein by its different rotatory power, and the names of α-casein and β-casein are therefore suggested for the casein and albumin respectively. Both of these caseins are soluble in the same proportions in the different constituents of the serum. Estimations of the solubility of α-casein in solutions of the serum constituents of approximately the same concentration as found in milk serum gave the following results: In lactose (5 per cent.), 0·27; sodium chloride (0·2 per cent.), 0·605; sodium citrate (0·08 per cent.), 0·945; and sodium phosphate (0·05 per cent.), 1·636 grms. per litre. This artificial serum would thus contain 3·456 grms. of α-casein; and assuming that β-casein dissolves in the same proportion, the total amount of caseins dissolved would be 6·9 grms. per litre, or somewhat higher than the average amount usually found (6·45 grms.). If a certain quantity of a solvent (*e.g.*, sodium citrate) be added to milk before coagulation, a serum richer in soluble caseins will be obtained (*e.g.*, 8·94 grms., instead of 6·12 grms. per litre for a 0·4 per cent. solution of sodium citrate). This increase is entirely due to the α-casein. In ordinary serum there is not more β-casein present than can be dissolved by the serum constituents, whereas only a part of the α-casein (about 10 per cent.) is in solution, the remainder being in a state of colloidal suspension. The addition of calcium chloride to boiled milk causes the latter to be coagulable by rennet. This is to be attributed to the salt precipitating a portion of the soluble caseins which have not been coagulated by the heat, and also to the precipitation of calcium phosphate, which mechanically envelops the casein in colloidal suspension. The coagulation of raw milk is also rendered more rapid and complete, and the coagulum firmer by the addition of calcium chloride, owing to the fact that part of the soluble caseins is rendered insoluble through the loss of the solvent constituents and the mechanical action of the nascent acid calcium phosphate.

C. A. M.

Nephelometric Determination of Proteins; Casein, Globulin and Albumin in Milk. P. A. Kober. (*J. Amer. Chem. Soc.*, 1913, **35**, 1585-1593.)—The author describes an improved form of nephelometer, and the following method of freeing a sample of milk from fat previous to casein estimation is recommended. Five c.c. of the milk are measured into a 250 c.c. graduated flask, and after adding 200 c.c. of water and 10 c.c. of $\frac{N}{10}$ caustic soda solution, water is added to the mark and the whole is thoroughly shaken. Ten c.c. are placed with 2 c.c. of ether (previously washed with 10 per cent. caustic soda solution) in a centrifuge tube, which is then tightly corked and vigorously shaken. After standing till the layers have separated, or centrifuging for a minute or so, 5 c.c. of the aqueous layer are removed with a pipette in such a way as not to disturb the ether, and diluted to 50 c.c. Milk so treated is quite clear and ready for nephelometric estimation. Taking 10 c.c. of this solution and adding 10 c.c. of 3 per cent. sulphosalicylic acid, a suspension of casein is obtained which can be matched with the following standard:—5 c.c. of a 0·01 per cent. casein solution mixed with 10 c.c. of 3 per cent. sulphosalicylic acid. The protein so obtained is not all casein, so another portion of milk is precipitated according to the "official" method (dilute acetic acid), and the amount of precipitate obtained in an aliquot portion of the filtrate is then determined nephelometrically by adding four volumes of the reagent. This fraction, called the "globulin and albumin" fraction, when deducted from the gross casein, gives the amount of casein precipitated by the "official" method. Results by the new method agree closely with the official figures, the former, if anything, being a trifle the higher. The ether used in extracting the fat increases the volume of the solution, so for 10 c.c. of diluted milk and 2 c.c. of ether, the factor 0·910 must be employed. For removing fat, hydrochloric acid cannot replace caustic soda, as this acid interferes with the use of sulphosalicylic acid. The standard casein solution must be controlled by a nitrogen estimation, using the factor 6·38, and is made by dissolving 0·1 grm. casein, or its equivalent, in 1 c.c. of $\frac{N}{10}$ caustic soda and making up to 100 c.c. with 2 c.c. toluene. This stock solution keeps for three or four days, and is diluted ten times for use. It is suggested that proteins may be directly estimated nephelometrically in potable waters. H. F. E. H.

Precipitation of Purine Bases from Meat Extract and Urine by Means of Zinc Salts. E. Salkowski. (*Biochem. Zeitsch.*, 1913, **55**, 254-260.)—On treating a 10 per cent. meat extract solution with ammonia and calcium chloride to remove phosphates, and adding the neutralised filtrate with zinc chloride and sufficient sodium carbonate to make the mixture neutral or faintly alkaline, a considerable precipitate is formed, which is easily filtered and washed. After removing the zinc from this precipitate by means of sulphuretted hydrogen, a solution is obtained in which purine bases can be scarcely detected by ammoniacal silver nitrate. This apparently negative result is due to the presence of colloidal substances which prevent the formation of the purine precipitate. That the zinc precipitate contains purine bases is proved by dissolving the precipitate in hot dilute nitric acid, with the addition of urea, cooling, and adding silver nitrate, when a precipitate of hypoxanthine silver or a mixture of this substance with hypoxanthine silver nitrate is formed. The presence of purine bases does not interfere with the determination of albumose nitrogen by means of zinc sulphate and sulphuric acid. E. W.

Estimation of Sulphur and Chlorine in the Rice Plant. A. R. Thompson. (*J. Amer. Chem. Soc.*, 1913, **35**, 1628-1634.)—Sulphur is found in plants in definite chemical organic compounds such as edestin (0·88 per cent. SO_3), legumin (0·38 per cent.), and gliadin (1·03 per cent.), as well as in the mustard oils. No organic compound containing chlorine has been isolated from any plant. From the obvious increase of sulphate ion in rice plants grown in sand and fertilised with ammonium sulphate, it is concluded that this salt as such is absorbed by the plant. Total sulphur was determined by Hart and Peterson's modification of Osborne's method, the melt being boiled out with water and not acid (*J. Amer. Chem. Soc.*, 1902, **24**, 142; *Wis. Agri. Expt. Sta. Res. Bull.*, **14**, 2). It is important that both fusions and boilings should be carried out over alcohol lamps. Duplicate determinations usually agreed within 0·02 to 0·03 per cent. Inorganic sulphur was determined by stirring the sample with 100 c.c. of 1 per cent. hydrochloric acid, and allowing it to stand an hour before filtration. Chlorine was determined by the Volhard official method (*U.S. Dept. of Agri. Bur. Chem. Bull.*, **107**, 23). Determinations of chlorine, total SO_3, and mineral SO_3 were made at three separate stages of growth of the plant, samples being taken both from unfertilised plots and from ground receiving a mixture of ammonium sulphate, superphosphate, and potassium sulphate. *First period*, before flowering, sample being divided into foliage and roots. In this series the total sulphur is higher than at any other period of growth, and is equally divided between foliage and roots. Inorganic sulphur is highest in the roots, being almost four times as high as in the foliage, but is still much lower than the total sulphur. Chlorine is also highest at this period, and, like sulphur, is found in highest percentage in the roots. *Second series*, taken at the flowering period. The sample was here divided into panicles, leaves, stems, and roots. The difference between manured and unmanured plants is nil as regards composition, though the former were much the larger and heavier plants, and the leaves show the highest, and the stems the lowest, content of sulphur. Chlorine, like sulphur, has decreased in percentage amount, and is highest in the roots. *Third series*, taken at maturity, and divided into chaff, grain, leaves, stems, and roots. Again no effect upon composition is shown by manuring, total sulphur (as SO_3) being very small in the stems and chaff, and about 0·23 per cent. on dry weight in the other parts. Inorganic sulphur is found only in the roots, showing that this form of sulphur has been used up in the formation of organic sulphur compounds. Chlorine is absent from the grain, though high in the stems. The pounds per acre of inorganic sulphur, phosphoric acid, and chlorine absorbed by the rice plant are as follows:

	Unmanured.	Complete Manure (as above).
SO_3	9·5	11·9
Cl	14·9	21·1
P_2O_5	28·8	42·0

An analysis of the soil showed that the sulphur is present largely in organic form; no trace of sulphide was detectable. The absence of effect of sulphur fertiliser on the composition of the plant is probably due to the presence of much iron in the soil, with the consequent formation of insoluble basic compounds. In sand pot experiments the chlorine absorbed is about twice as great as that absorbed by the rice plant when grown under normal conditions, experiments by the sand method showing that sulphur fertiliser is not rendered insoluble to the plant, as is the case in soil cultures. H. F. E. H.

Detection of Albumin in Urine. O. Mayer. (*Zeitsch. angew. Chem.*, 1913, **26**, 639-640.)—Several tests should be made in order to be certain that any given sample of urine contains albumin. Ten c.c. of the urine should be boiled with 5 c.c. of sodium chloride solution (1 : 3) acidified with acetic acid; a precipitate indicates the presence of albumin. Another portion of the urine is mixed with an equal volume of a reagent prepared by dissolving 4 grms. each of mercuric chloride and sodium chloride and 8 grms. of citric acid in 250 c.c. of 6 per cent. acetic acid; a white turbidity is obtained when the urine contains albumin. Mucin is also precipitated by this reagent, and a preliminary test should be made by boiling the urine with an equal volume of acetic acid, when any turbidity produced will be due to mucin. A comparison of the two turbidities will show whether albumin is present in addition to the mucin. The quantity of albumin present in urine may be estimated approximately by allowing 5 c.c. of the sample to flow on to the surface of about 10 c.c. of a solution containing 5 grms. each of mercuric chloride and citric acid and 40 grms. of sodium chloride in 500 grms. of water. When the urine contains albumin to the extent of 0·001 per cent. a white ring forms in ninety seconds at the junction of the two liquids. Should the ring appear in a shorter time, the urine is diluted with water and the test repeated until the ring develops in the time mentioned; the amount of albumin present is obtained from the degree to which the urine has been diluted. W. P. S.

Estimation of *B. Coli Communis* in Water. J. Partiš. (*Arch. f. Hyg.*, 1913, **79**, 301-322; through *Chem. Zentralbl.*, 1913, II., 1252-1253.)—A measured quantity of the water to be tested is placed in suitably graduated centrifugal tubes; with ordinary not highly polluted waters, 40 or 20 c.c. may be taken, but with highly polluted samples and surface waters, 10 or 5 c.c. will suffice. Alternatively, the water may be diluted with physiological sodium chloride solution, and a suitable quantity measured off. If 40 c.c. be taken, in the case of soft waters, 20 drops of sterile 10 per cent. sodium carbonate solution and 10 drops of sterile 10 per cent. ferric sulphate solution are added; with hard waters, 16 and 8 drops respectively are employed. The tubes are centrifuged for ten minutes, and the clear liquid is carefully poured off from the precipitate. The latter is dissolved in 1 c.c. of sterile, neutral, 20 per cent. potassium tartrate solution, the liquid is thoroughly mixed and distributed quantitatively among six to twelve Conradi-Drigalski plates, according to the suspected degree of pollution of the water. The walls of the tube are rinsed out with the solvent and the washings also brought on to the plates. The plates, without covers, are first digested for thirty to forty-five minutes in the thermostat at 43° to 45° C. and then kept at 37° C. When the red colonies are sufficiently developed, they are counted, and a few are examined in the usual way to confirm their identity as typical *B. coli.* The method described is sensitive and permits of the collection and enumeration of all the *coli* germs in a measured quantity of water; the colonies develop on the plates in twenty-four hours, and the organisms are available for further cultural and biological tests. By this method also, other pathogenic organisms of the typhus-coli group, which may be present in the water, can be detected.

J. F. B.

Bacteriological Methods of Water Analysis, with Special Reference to the Use of the Berkefeld Filter. E. Hesse. (*Arch. f. Hyg.*, 1913, **80**, 11-28; through *Chem. Zentralbl.*, 1913, II., 1253.)—The author gives a summary of the various methods for counting organisms and the detection of pathogenic bacteria and *B. coli.* The methods which depend on the employment of the Berkefeld filter and the bacteriological examination of the residue retained by the filter are specially dealt with. The candles supplied by the Berkefeld Filter Company, under the designation "$10\frac{1}{3}$," are particularly suitable; owing to their larger surface, these filter much more rapidly than the No. 12 candles. Without the use of kieselguhr, 42 per cent. of the organisms present in the water are recovered, but with kieselguhr 91 to 97 per cent. can be retained. A further advantage is that large quantities of the water to be tested can be employed.

J. F. B.

ORGANIC ANALYSIS.

Detection of Amyl Alcohol and other Aliphatic Alcohols and their Esters. T. Takahashi. (*J. Coll. Agric. Tokio*, 1913, **5**, 167; through *Chem. Zentralbl.*, 1913, II., 1334.)—In elaboration of his method (*Bull. Coll. Agric. Tokio*, **7**, 437) and that of Koshino (*Pharm. Soc. Jap.*, No. 351) for the detection of amyl alcohol (fusel oil) with vanillin and sulphuric acid, the author gives the following method: 2 c.c. of a solution of vanillin in 200 c.c. sulphuric acid of sp. gr. 1·84, with 0·5 c.c. of 0·1 per cent. amyl alcohol (Merck) in 15 per cent. alcohol, give a red coloration which increases in intensity until finally the liquid becomes turbid. Water added in drops clears the solution and gives a purple coloration which becomes bluish on the further addition of water, up to 4 c.c. Within ten days a precipitate of similar colour forms, while the supernatant liquid is a light rose-red. In a similar manner the following colours were obtained with other alcohols: Absolute ethyl alcohol (1 c.c.), turbid greenish-blue—light green; methyl alcohol, dark red—purple; propyl alcohol, purplish-red—red—purplish-blue; isopropyl alcohol, purplish-red—red—purple; *n*-butyl alcohol, yellow—red—purple; amyl acetate, red—vermilion—carmine; amyl formate, light yellow—red—purplish; amyl benzoate, yellow—purplish-red; butyl ester, green—pink; amyl ester, red—light purplish-red—purple; butyl isovalerate, red—reddish-yellow—vermilion—purplish-red; propyl valerate, light red—carmine—purplish-blue; acetic ester, dark yellow—light pinkish-red; methyl isobutyrate, light red; methyl propionate, red—purplish-red—purple; methyl lactate, green—dark green—red; methyl acetate, purplish rose-red; methyl formate, light red; ethyl propionate, light red; caproic ester, yellow—carmine-red; aceto-acetic ester, green—greenish-red—red (blue precipitate). In all cases but the first, 1 drop of the alcohol or ester was taken. O. E. M.

Estimation of Small Quantities of Methyl and Ethyl Alcohols in Aqueous Solutions. J. Hetper.

(*Zeitsch. Untersuch. Nahr. Genussm.*, 1913, **26**, 342-348.)—Methods described previously by the author (ANALYST, 1912, **37**, 366; 1913, 56), and based on oxidation with permanganate, may be applied to the estimation of ethyl and methyl alcohols in dilute solution. The solution must not contain other substances which reduce permanganate, and previous to the estimation it must be diluted with water or concentrated by distillation, as the case may be, until it contains from 0·1 to 0·25 per cent. by weight of total alcohols. The solutions required are: Acid $\frac{N}{2}$ potassium permanganate solution prepared by dissolving 16 grms. of potassium permanganate and 40 grms. of crystallised phosphoric acid in 1 litre of water, and alkaline permanganate solution containing 16 grms. of the salt and 40 grms. of sodium hydroxide per litre; these two solutions are standardised against $\frac{N}{2}$ oxalic acid solution. A preliminary experiment is made to determine the alcoholic strength of the solution under examination; for this purpose, 25 c.c. of the alkaline permanganate solution are diluted with 50 c.c. of water, heated on a water-bath, and the alcoholic solution is added in small quantities at a time until the permanganate colour is changed to green. The mixture is heated for a further twenty minutes, 25 c.c. of $\frac{N}{2}$ oxalic acid solution are added, and the excess of the latter is titrated with permanganate. Each c.c. of $\frac{N}{2}$ permanganate solution is equivalent to 0·0025 grm. of alcohol.

Alkaline Oxidation.—Twenty-five c.c. of the alkaline permanganate solution are diluted, heated to 40° C., 10 c.c. of the alcohol solution are added, and the mixture is at once heated on a water-bath, where it is maintained at 94° C. for from fifteen to twenty minutes. The hot solution is then treated with 25 c.c. of $\frac{N}{2}$ oxalic acid solution, and titrated with the acid permanganate solution. The quantity of permanganate solution reduced should be not less than 7 c.c. or more than 12 c.c.

Acid Oxidation.—Thirty c.c. of the acid permanganate solution are diluted with water, from 10 to 20 c.c. of the alcohol solution are added, and the mixture is heated under a reflux condenser to 94° C. for two hours. Thirty c.c. of $\frac{N}{2}$ oxalic acid solution are then added, and the excess is titrated with permanganate solution. The volume of permanganate solution reduced should be from 8 to 14 c.c., and 0·1 c.c. is deducted from the quantity used.

Let $p =$ grm. of total alcohols in the solution, $x =$ grm. of methyl alcohol in 100 grms. of total alcohols, $y =$ grm. of methyl alcohol in 100 c.c. of the solution, $z =$ grm. of ethyl alcohol in 100 c.c. of the same, $n_1 =$ c.c. of alcoholic solution, and $v_1 =$ c.c. of $\frac{N}{2}$ permanganate solution used in the alkaline oxidation, $n_2 =$ c.c. of alcohol solution, and $v_2 =$ c.c. of $\frac{N}{2}$ permanganate solution used in the acid oxidation, then:

$$p = \frac{0·2667\ v_1}{n_1}, \qquad x = \frac{50\ v_2}{n_2\ p} - 87, \qquad y = \frac{px}{100},$$

$$x = p - \frac{px}{100} \text{ or } \frac{\frac{v_1}{n_1} - \frac{v_2}{n_2}}{2·01}$$

W. P. S.

Simultaneous Estimation of Methyl Alcohol and Formaldehyde. M. Nicloux. (*Bull. Soc. Chim.*, 1913, **13**, 935-939.)—The method is a modification of the author's dichromate process (ANALYST, 1898, **23**, 41). The amount of potassium dichromate required for the oxidation of the mixed methyl alcohol and formaldehyde is first estimated, and then in a second estimation the quantity of carbon dioxide produced in this oxidation. For the latter estimation, 10 c.c. of the solution are mixed with sufficient 2N-dichromate solution to give a green tint, and the mixture placed in a small tube which is introduced upright into a reaction vessel containing 15 c.c. of sulphuric acid. The small tube is inverted within the flask after reduction of pressure, and the reaction completed by immersing the apparatus for five minutes in an oil bath at 150° C. The gas is then extracted by means of a mercury pump and measured. If x represent the required quantity of methyl alcohol, y the formaldehyde, and a the weight of carbon dioxide produced by the oxidation of 5 c.c. of the mixture by the quantity b of dichromate—

$$\frac{44x}{32} + \frac{44y}{30} = a,$$

and

$$\frac{294x}{32} + \frac{196y}{30} = b,$$

since 32 mgrms. of methyl alcohol require 294 mgrms. of potassium dichromate for oxidation, and yield 44 mgrms. of carbon dioxide; whilst 30 mgrms. of formaldehyde require 196 mgrms. of dichromate, and yield 44 mgrms. of carbon dioxide.

Hence

$$x = \frac{16\ (11b - 49a)}{539},$$

and

$$y = \frac{15\ (147a - 22b)}{1078}.$$

C. A. M.

Coals of Canada. (*Bull. Imp. Inst.*, 1913, **13**, 496-513.)—Analyses of the coals from all the Canadian coalfields are given. C. A. M.

Analysis and Examination of Non-inflammable Cellulose Esters. Acetylcelluloses. H. Barthelemy. (*Monit. scient.*, 1913, **3**, II., 549-554; through *Chem. Zentralbl.*, 1913, II., 1617.)—The following tests may be applied for the examination of cellulose acetate preparations: Ten grms. of the sample are digested for one or two days, with frequent shaking, with a mixture of 190 grms. of dry tetrachloroethane and 10 grms. of 96 to 98 per cent. methyl alcohol, and the solution is examined for insoluble residue; the strength and transparency of the film left by the evaporation of the solvent are also noted. From the relative viscosity of the solution an idea may be obtained as to the extent of degradation suffered by the cellulose molecule in the process of acetylation. The presence of admixtures of nitro-cellulose may be detected by the diphenylamine-sulphuric acid test. The moisture is determined by heating 10 grms. of the powdered material at 65° to 70° C. until the weight is constant. A good acetate should not lose more than 1 to 1·5 per cent. under such conditions. The ash should not exceed 0·4 to 0·6 per cent. The percentage of combined acetic acid is estimated by saponification of 2 grms. of the material by heating for sixteen hours with 40 c.c. of $\frac{N}{1}$-sodium hydroxide solution at 85° to 90° C., and titrating back the excess of alkali with $\frac{N}{5}$ sulphuric acid. The "copper value" (cupric reducing value) is of special importance from the point of view of the practical utility of the acetate; it is inversely proportional to the toughness and viscosity of the material, and should not exceed 10. For testing the stability, 5 grms. of the powdered acetate are heated in a slow current of air free from carbon dioxide for three hours at 123° to 125° C., the air then passing through a flask containing $\frac{N}{50}$ sodium hydroxide solution, which is afterwards titrated back. The "instability value" (grms. of acid, calculated as acetic, split off per 100 grms. of material) should not exceed 0·3. J. F. B.

Methods for the Estimation of Natural Gas for the Production of Gasoline. E. S. Merriam and J. A. Birchby. (*J. Ind. and Eng. Chem.*, 1913, **5**, 824-828.)—Two empirical tests are described and recommended, and a small testing compressor is illustrated by means of which a direct determination can be carried out. The first test consists in shaking 1 c.c. of kerosene with two successive portions of 100 c.c. of the gas. The shaking is carried out in a Hempel explosion pipette over mercury, and the residual gas measured in each case. The actual volume absorbed from the second 100 c.c. as well as the ratio of the volumes absorbed in the two experiments is significant. As a result of the first shaking, the kerosene is saturated with methane, which is usually present in large amount, and is not very soluble, and nearly saturated with ethane, but is very far from being saturated with the higher paraffins, which are present in smaller amount and freely soluble. The decrease in volume in the second experiment is therefore mainly due to the higher paraffins, and the number of c.c. absorbed gives approximately the number of gallons of gasoline which can be obtained from 1,000 cubic feet of gas. When this number is high (5 or more) the ratio of gas absorbed in the first experiment to that absorbed in the second is usually low (2 or thereabouts), whereas when the proportion of liquid hydrocarbons is low (less than 1 gallon per 1,000 cubic feet) the said ratio may run up to 10.

A second experiment, which gives useful results, is the following: A measured volume of gas (100 c.c.) is passed backwards and forwards between a gas burette and a Hempel pipette through a tared bulb tube containing 5 c.c. of olive oil, and from the contraction in volume of the gas and increase in weight of the bulb tube the mean molecular weight of the dissolved hydrocarbons is calculated. The olive oil is saturated with methane and ethane before use. Gases containing paying quantities of gasoline (1 gallon per 1,000 cubic feet and upwards) yield hydrocarbons to olive oil with a mean molecular weight of 60 to 73, whereas gases containing only small proportions of liquid hydrocarbons give a figure of 48 to 60 when submitted to this test.

The third test described is a direct determination of the liquid condensate which can be obtained from 250 cubic inches of gas. The apparatus consists of a pair of brass cylinders, each provided with a piston, together with a measuring cylinder, the whole tested to 500 pounds working pressure. It is operated by a small boiler-testing pump. Pressures from 80 to 250 pounds per square inch are used in actual large-scale plant for the recovery of gasoline from natural gas. G. C. J.

Colorimetric Method for the Estimation of Histidine. M. Weiss. (*Chem. Zeit.*, 1913, **37**, 1245.)—The method depends on the property which histidine possesses of combining with diazotised compounds to form a red-coloured substance. The reagents required are: (1) Sulphanilic acid, 4, hydrochloric acid, 40, water, 400 parts; (2) 0·5 per cent. sodium nitrite solution; (3) 10 per cent. sodium hydroxide solution. One volume of the sulphanilic acid solution is added to 2 volumes of the nitrite solution to form the diazotised compound. A standard is prepared by treating 10 c.c. of a 0·01 per cent. histidine solution with 1·5 c.c. of the diazotised reagent and 3 c.c. of the sodium hydroxide solution, and histidine solutions under examination are treated similarly, if necessary, after dilution to such an extent that the coloration obtained exactly matches the standard. Tyrosine must not be present in the histidine, its absence being denoted by a negative reaction with Millori's test. Histidine sometimes contains other substances which interfere with the diazo reaction, since they combine with a portion of the reagent. It is recommended that portions of the solution be treated with increasing quantities of the reagent until an optimum coloration is obtained, the comparisons being made after the standard and the test solution have been diluted to a volume of 20 c.c. each. W. P. S.

New Indican Reaction. A. Jolles. (*Chem. Zeit.*, 1913, **37**, 1200.)—The test depends on the formation of a violet-coloured compound when thymol and an oxidising agent are brought into contact with potassium indoxylsulphonate or with the indoxyl liberated from the latter by means of hydrochloric acid. In applying the test to urine, 10 c.c. of the sample are mixed with 2 c.c. of a 20 per cent. lead acetate solution and filtered; the filtrate is then treated with 0·5 c.c. of a 10 per cent. alcoholic thymol solution, 10 c.c. of hydrochloric acid containing ferric chloride

(Obermayer's reagent), and 4 c.c. of chloroform, and the mixture is shaken. The presence of a mere trace of indican produces a violet coloration in the chloroform layer. When shaken with water the colour changes to brownish-yellow, but is reproduced on shaking the chloroform layer with hydrochloric acid. It may happen that when thymol has been added to urine as a preservative, the usual tests for indican may yield a violet or blue coloration; this may be differentiated by the ordinary starch test from the colour given by iodine. W. P. S.

Colour Reactions of Triketohydrindene Hydrate (Ninhydrin). W. Halle, E. Loewentstein, and E. Pribram. (*Biochem. Zeitsch.*, 1913, **55**, 357-370.)—A number of substances containing primary alcoholic, aldehydic, and ketonic groups give colour reactions on boiling with ninhydrin; thus glycerol, amyl alcohol, and dextrose give blue colorations, ethylene glycol and maltose bluish-violet, lævulose and lactose reddish-violet, erythritol red, and galactose rose colours. These reactions occur only in highly concentrated and neutral solutions, and do not take place in dilute solutions or in the cold. The sensitiveness of the reaction is greatly increased if the liquid is made alkaline after boiling: sodium, calcium, and barium hydroxides giving blue, ammonia red, and sodium carbonate violet-red colorations. In the case of dextrose, a distinct blue colour is produced on adding sodium hydroxide after boiling with ninhydrin even at a dilution of 1 in 100,000. The reaction consists in the reduction of the triketohydrindene hydrate to hydrindantin, which substance is an exceedingly sensitive indicator for free hydroxyl ions. The formation of blue colour on boiling ninhydrin with amino-acids differs from that described above, in that it occurs in the cold and even *in vacuo*, and is not increased by subsequent addition of free hydroxyl ions, whilst addition of sodium hydroxide before boiling prevents the reaction. Hydrindantin also gives a blue colour with amino-acids in the cold. The colour formed from ninhydrin with amino-acids is colloidal, and especially sensitive towards electrolytes, the action depending almost exclusively on the nature of the cation. E. W.

Solidifying and Melting-Points of Mixtures of Stearic and Oleic Acids. R. Meldrum. (*Chem. News*, 1913, **108**, 199-201.)—Parallel determinations of the solidification-point of prepared mixtures of commercial "stearic" and "oleic" acids by Dalican's method showed that the results were accurate to within 13·3 per cent. of the solid fatty acid. The error decreased with the proportion of oleic acid present, so that the degree of accuracy with a mixture containing 20 per cent. of "stearic" acid was 11·4 per cent. Thus, whilst the addition of 10 per cent. of oleic acid only lowered the solidification-point by 1·5° C., the addition of 80 per cent. caused a lowering of 0·61° C. for each 1 per cent. of liquid fatty acid present. Again, in the case of a mixture containing 70 per cent. of oleic acid, each 1 per cent. of the latter corresponded to a lowering of 0·46° C. Similar results, but correspondingly lower, were obtained when the determinations were made without stirring the mixture. The final rise of the temperature varied as much as 0·5° C. in a series of determinations. When working with large quantities of fatty acids, the "rise" was apparently eliminated owing to the slowness of cooling. As regards the melting-point, the most constant results were obtained by the thermometer bulb method, care being taken that the coating of fatty acid was of uniform thickness. The conclusion drawn from determinations by four methods is that the results obtained by all the methods are too high, and that for this reason the solidifying-point is only apparently, and not really, lower than the melting-point. C. A. M.

"Wood Oils" from China and Japan. E. H. Wilson. (*Bull. Imp. Inst.*, 1913, **13**, 441-461.)—About nine-tenths of the wood oil of commerce is derived from *Aleurites Fordii*, and is exported from Hankow. A sample of the seeds of this tree yielded 58·3 per cent. of oil with sp. gr. 0·940 at 15° C., saponification value, 191·8, and iodine value, 166·7. Chinese wood oil is also obtained from another species, *A. montana*. The Japanese oil is derived from *A. cordata*, which has frequently been confused with the two Chinese species. The Japanese oils examined by Chapman (ANALYST, 1912, **37**, 551) were stated to be the product of *Paulownia imperialis*, but later examination has identified the seeds from which these oils were obtained as those of *Aleurites cordata*. The Japanese and Chinese oils differ in polymerising properties, the former remaining fluid under conditions which convert the latter into a hard jelly which becomes a friable powder when crushed between the fingers. C. A. M.

Waxes and Resins of Kapok and Akon Fibres. H. Matthes and L. Streicher. (*Arch. Pharm.*, 1913, **251**, 438-452.)—When extracted with a mixture of alcohol and benzene, kapok fibres yielded 4·97 per cent., and akon fibres 4·63 per cent. of waxes resembling beeswax. These waxes contained 28 and 31 per cent. respectively of unsaponifiable matter (mainly phytosterol and melissyl alcohol) and gave the following values:

	$[N]_{D\ 40^\circ\ C.}$	Melting-Point.	Acid Value.	Saponification Value.	Iodine Value.	Reichert-Meissl Value.	Polenske value.
Kapok wax	1·4618	24° C.	58·85	170·14	69·44	2·02	0·97
Akon wax	1·4682	30·5° C.	65·09	171·52	70·52	1·76	1·05

The fatty acids of kapok wax consisted of about 15 per cent. of solid acids and 85 per cent. of liquid acids, whilst the corresponding amounts in akon wax were 20 and 80 per cent. respectively. In both cases the solid acids consisted of palmitic acid, whilst the liquid fatty acids contained about 61 per cent. of oleic acid, 38 per cent. of linolic acid and 1 per cent. of linolenic acid. Both kapok and akon fibres contained bitter principles, and from the latter a resinous substance resemblng aloes resin was isolated. C. A. M.

INORGANIC ANALYSIS.

Quantitative Ammonia Distillation by Aëration. III. P. A. Kober and S. S. Graves. (*J. Amer. Chem. Soc.*, 1913, **35**, 1594-1604.)—In two previous papers (ANALYST, 1908, **33**, 409, 442), P. A. Kober dealt with the aëration method of distilling ammonia, and the authors now elaborate the method with further details, and cite numerous experiments showing its accuracy and convenience for all classes of ammonia determinations. From 400 to 600 litres of air were found to be sufficient for the average amount of ammonia obtained in Kjeldahl estimations. The heat of neutralisation, though slightly accelerating the distillation at the beginning, has no effect on the final results. The best quantity of sodium hydroxide is found to be 75 c.c. of a solution of sp. gr. 1·46 to 1·48, and 20 c.c. of concentrated sulphuric acid. Full details, with an illustrated apparatus, are given for the best method of manipulating the aëration of the Kjeldahl melt after digestion. Using a fairly good pump, 25 c.c. of $\frac{N}{10}$ ammonia can be quantitatively distilled (by aëration) in about one hour. Litmus-paper is not suitable for the detection of small amounts of ammonia in distillates if used in the ordinary way, but should be allowed to soak with stirring until change of colour is apparent. Boric acid, suggested by Winkler in place of the usual standard acid as an absorbent for the ammonia, is not satisfactory if used for the aëration method (*cf.* ANALYST, 1910, **35**, 316). H. F. E. H.

Electrolytic Deposition of Antimony. W. K. Chaney. (*J. Amer. Chem. Soc.*, 1913, **35**, 1482-1490.)—The paper is concerned with the deposition of antimony in presence of tin. In absence of tin, it may be deposited rapidly and quantitatively from an electrolyte of sodium sulphide, potassium cyanide, and sodium hydroxide. This method, as well as the method of depositing tin from the solution of its sulphide in ammonium polysulphide, breaks down when both metals are present, as tin is incompletely deposited from a sodium sulphide electrolyte, and metallic antimony is appreciably soluble in ammonium sulphide. The author has worked out an entirely satisfactory method for the simultaneous deposition of tin and antimony, and another method which, with care, effects the deposition of antimony alone from a solution containing both metals.

The simultaneous deposition of antimony and tin is effected from an ammonium polysulphide electrolyte under conditions that insure that at the end of the electrolysis the concentration of ammonia and ammonium sulphide is negligible. The initial concentration of ammonium polysulphide is also important. Concentrated aqueous ammonia is saturated with hydrogen sulphide in presence of excess of sulphur, and the filtered solution is mixed with six times its bulk of ammonia, saturated with hydrogen sulphide. This mixture is added drop by drop to the solution of the chlorides of antimony and tin until a clear solution is obtained. This solution is then diluted to about 125 c.c. in a platinum dish, which serves as cathode. An anode rotating at 400 revolutions per minute is used, and the solution electrolysed at the boiling-point with an initial voltage of 3·5 and $N.D_{100} = 1$ amp., raising the voltage to 4 as the current falls. From 0·2 to 0·3 grm. of antimony may be deposited quantitatively within an hour. At the boiling temperature ammonia and ammonium sulphide are removed or oxidised, and the deposits are like polished platinum.

Antimony may be separated from tin as follows: The solution of the mixed chlorides or the suspension of the mixed sulphides in water is treated with hydrogen chloride and boiled. Excess of powdered lime is added, and the mixture is again boiled. Some lime is suspended in boiling water, the mixture saturated with hydrogen sulphide, filtered, and the filtrate added to the contents of beaker containing the sulphides of tin and antimony. The mixture is boiled for ten minutes, allowed to cool to 70° C., and electrolysed at 2·5 volts and $N.D_{100} = 0·2$ amp., using a rotating cylindrical platinum cathode 12·5 cm. in circumference and 4 cm. in height, and an anode of platinum iridium wire bent to form a helix, 6 cm. in diameter, outside the cylinder, and then to form a smaller (1 cm.) helix inside the cylinder. The success of the method depends on getting the stannic hydroxide precipitated in a compact form. If in a gelatinous form, particles may adhere to the cathode and become reduced. G. C. J.

Calorimetric Ash Estimations. F. v. Konek-Norwall. (*Chem. Zeit.*, 1913, **37**, 1181-1182.)—The ash remaining in the crucible or other contrivance which accommodates the charge in an oxygen calorimeter is always less than the ash obtained by burning the substance in an ordinary open dish. This is known to be partly due to scattering of the charge and partly to other causes. The author finds that the error is particularly large with coals high in sulphur, and may amount to one-third of the ash with Hungarian coals, with 5 per cent. of sulphur. In such cases notable quantities of sulphate of calcium, iron (and the alkali metals), are found in the bomb washings, and the author recovers the lime and ferric oxide by filtering off the precipitate which results from the titration of the bomb washings with sodium carbonate. The washed precipitate is ignited with the paper, and the weight of the ash added to that remaining in the bomb crucible. Even when so corrected, the results are always below the truth, one source of loss being due to alkali oxide, which is lost as sulphate. G. C. J.

Estimation of Chlorine in Rag Flock. A. E. Parkes. (*Chem. News*, 1913, **108**, 177.)—Fifty grms. of a representative sample are digested for three to four hours with 500 c.c. of water, and the extract filtered through wire gauze in a Buchner's funnel. The residue is washed with successive portions of 100 c.c. of water, until the filtrate and washings amount to 1,000 c.c. This extract is filtered through paper, and portions of 100 c.c. (=5 grms. of flock) are evaporated to dryness in porcelain dishes, and the residues heated until the organic matter is just charred, after which water is added and the chlorine titrated with silver nitrate solution, with potassium chromate as indicator. The method gave accurate and concordant results in the case of samples containing known quantities of sodium chloride. In examining unwashed flocks the addition of pure phenol or other germicide to the first extract is advisable (*cf.* ANALYST, 1913, 469). C. A. M.

Estimation of Copper as Sulphide. C. Beck. (*Chem. Zeit.*, 1913, **37**, 1330.)—For the reduction of cupric sulphide to cuprous sulphide by ignition with sulphur in a Rose crucible, a current of carbon dioxide may with advantage replace the current of coal gas or hydrogen usually employed. The advantage lies in the fact that there is no risk of reduction to metallic copper whatever the temperature employed, and no risk of explosion or of fracture of the crucible lid due to local heating where the hydrogen, etc., escapes and burns. G. C. J.

Cathodic Estimation of Lead and Analysis of Lead Alloys. R. Gartenmeister. (*Chem. Zeit.*, 1913, **37**, 1281-1282.)—Hitherto lead has been estimated electrolytically only as peroxide on the anode. Conditions are now described under which it may be deposited quantitatively as metal on the cathode. For the estimation of quantities of lead up to 1 grm., the electrolyte should contain 2 to 2·5 c.c. concentrated nitric acid, 5 grms. gallic acid, 5 to 6 c.c. alcohol, and amount to about 125 c.c. For quantities of lead from 1 grm. to 2·5 grms., these quantities should be doubled. Larger quantities of lead than 2·5 grms. cannot be deposited free from impurities by the use of the electrodes described. These consist of a Winkler gauze cathode and a cylindrical anode of platinum foil, 7 mm. in diameter and 40 mm. long. The anode is centred within the cathode, so that the distance from any point on the anode surface to the nearest point of the cathode surface is almost a constant. The current should not deviate much from 1·2 amps. At 1·6 amps. a non-adherent deposit results, whilst a current less than 1·2 amps. makes the deposition very slow. The electrolyte is maintained at 65° to 75° C. throughout the process, which requires from three to five hours, according to the amount of lead present. The longer time suffices for the deposition of 2·5 grms. lead. The operation should not be prolonged unnecessarily if the most exact results are desired, as a small quantity of a yellow-coloured substance may then be deposited with the lead. The error involved in continuing the electrolysis for five hours with only 0·5 grm. lead present is about 1 mgrm. At the end of the operation the cathode is washed once with tap-water without interrupting the current; then with distilled water, alcohol, and ether; and is finally dried cautiously over a flame. In absence of interfering elements, quantities of lead from 0·4 grm. to 2·5 grms. can be estimated with an absolute error in no case exceeding 1·7 mgrms., and a percentage error in no case exceeding 0·13 per cent.

Copper, if present, is wholly deposited with the lead. Zinc, cadmium, iron, nickel, cobalt, and manganese remain wholly in the electrolyte. Silver, tin, antimony, and arsenic interfere with the method, as they are partially, but not wholly, deposited on the cathode. In the important case of alloys of lead with tin and (or) antimony, the metal is dissolved in nitric acid with the aid of tartaric or citric acid; the solution is made alkaline with solid sodium hydroxide, mixed with a concentrated solution of sodium sulphide, warmed, and filtered. The precipitate, which contains all the copper, iron, and zinc, as well as lead, is washed with sodium sulphide solution until free from tin and antimony, and then with a strong solution of ammonium chloride containing a little ammonia and ammonium sulphide. It is then dissolved in nitric acid as far as possible, and the filter, which obstinately retains some lead, is ashed, and the ash dissolved in nitric acid and added to the main solution, which is next evaporated to dryness. The residue is taken up in nitric acid and electrolysed as described. In the filtrate and washings containing the tin and antimony, the latter is estimated electrolytically by Hollard's modification of Classen's method. The tin in the exhausted electrolyte is precipitated as sulphide, and this is dissolved by means of hydrochloric acid and potassium chlorate, and the solution evaporated to dryness. The residue is dissolved in hydrochloric acid, and the tin deposited from an ammonium oxalate electrolyte by Hollard's modification of Classen's method. If the alloy contains tin or antimony, but not both, the method is simplified.

G. C. J.

Analysis of Alloys of Lead, Tin, Antimony, and Copper. D. J. Demorest. (*J. Ind. and Eng. Chem.*, 1913, **5**, 842-843.)—The novelty of the method here described lies mainly in the titration of antimony by permanganate in sulphuric acid solution free from hydrochloric acid. The combination of methods for determining the four metals has also points of novelty. The drillings (1 grm.) are treated with 20 c.c. hot concentrated sulphuric acid until the sample is nearly decomposed and the lead sulphate white. Free sulphur is liberated, but does not interfere. The mixture is boiled for some minutes, cooled, diluted with 50 c.c. water, boiled several minutes, and, before it has cooled to 60° C., the clear liquid above the lead sulphate is decanted through a Gooch crucible. The lead sulphate is boiled up with a further 10 c.c. of sulphuric acid, the mixture diluted with 30 c.c. water, boiled, and filtered through the Gooch crucible. The lead sulphate is washed with hot water, dried, ignited at a dull red heat, and weighed. The filtrate, which should not exceed 150 c.c., is heated to 60° to 80° C., and titrated with permanganate until a deep red colour appears. More permanganate (2 to 3 c.c.) is added, the solution shaken for a minute, and the excess of permanganate determined by titration with ferrous sulphate. The net consumption of permanganate gives the antimony. To the titrated solution 50 c.c. of hydrochloric acid and about 0·15 grm. antimony (unless this amount is already present) dissolved in 5 c.c. concentrated sulphuric acid are added, and the boiling solution is reduced by means of iron in a current of carbon dioxide, the boiling and stream of gas being continued for thirty minutes after the solution becomes colourless. The stannous solution is cooled in a current of carbon dioxide and titrated with iodine.

Another portion of the sample is treated in a similar manner up to, or including, the antimony titration. Tartaric acid (3 grms.) is added, and then ammonia, until the solution is slightly alkaline. The acid reaction is restored by addition of 2 c.c. sulphuric acid, the solution is heated nearly to boiling, 2 grms. sodium sulphite is added, and, when it has dissolved, 10 c.c. of a 10 per cent. solution of potassium thiocyanate. The mixture is shaken, allowed to stand fifteen minutes, filtered through asbestos, and the copper precipitate washed. Copper is then estimated either by the thiocyanate-permanganate method or by the iodide method after ignition of the precipitate and solution of the oxide in nitric acid.

The results are accurate to within 0·2 per cent. for lead, tin, and antimony, and to within 0·1 per cent. for copper.

G. C. J.

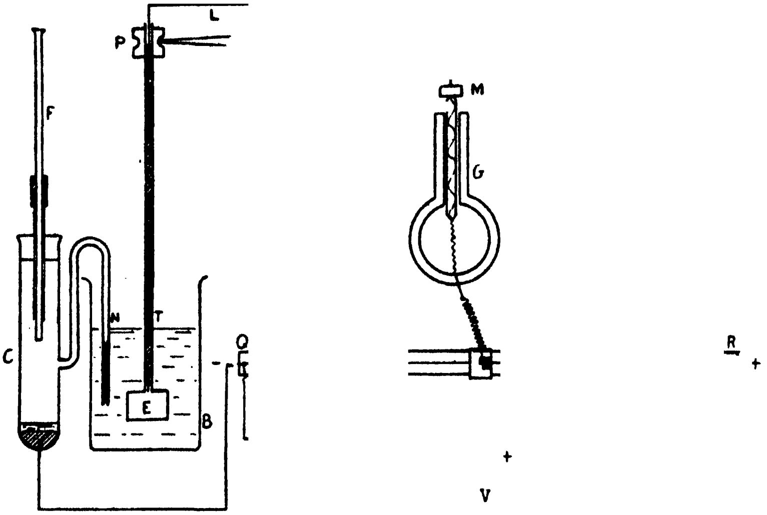
Spectroscopic Detection of Magnesium, Calcium, Strontium, and Barium. E. H. Riesenfeld and G. Pfützer. (*Ber.*, 1913, **46**, 3140-3144.)—By ordinary spectroscopic methods—for example, using the Beckmann burner—calcium cannot be detected in solutions of lower concentration than 0·2 mgrm. per c.c., strontium in concentrations below 0·6 mgrm. per c.c., barium in concentrations below 14 mgrms. per c.c., and magnesium not at all. By resort to an arc spectrum, far smaller quantities of calcium, strontium, and magnesium can be detected, and the detection of magnesium also falls within the range of this method. The authors figure a suitable apparatus, consisting of a glass cup to accommodate 1 to 2 c.c. of the liquid to be examined, the cup pierced by a platinum wire resting in an ebonite holder containing a little mercury, into which the wire dips. The mercury in the ebonite holder is in electrical connection with a brass binding screw at its base, the holder being suspended by a clamp which grasps it near the top. Above the liquid is suspended an iridium needle, adapted to be put in connection with the other terminal of the source of electrical energy. The whole apparatus is mounted on a stand facilitating vertical and horizontal adjustment of the position of the arc relative to the spectrometer. The liquid in the cup is connected to the positive pole, of a source of electrical energy at 100 to 200 volts. Between the negative pole and the iridium needle is interposed an adjustable resistance of 300 to 500 ohms, to carry a current of 1 amp. The iridium needle is depressed by means of a micrometer screw until an arc is established between it and the liquid, and the length and intensity of the arc are then regulated by further adjustment of the micrometer screw and of the external resistance. As little as 0·002 mgrm. calcium, 0·03 mgrm. strontium, 0·006 mgrm. barium, and 0·1 mgrm. magnesium, may be detected easily in 1 c.c. In these concentrations, calcium shows one brilliant line (423 $\mu\mu$), a bright line (616 $\mu\mu$), and a faint line between them; strontium two bright lines (422 and 461 $\mu\mu$) and two fairly bright lines; barium two brilliant lines (455 and 493 $\mu\mu$), two other bright lines, and a fairly bright one; and magnesium a brilliant band composed of three lines (516·8 to 518·4 $\mu\mu$), as well as a fairly bright line further towards the violet end of the spectrum. The iridium electrode gives rise only to lines which are faint compared with the above. The hydrogen line 656 $\mu\mu$ appears as a brilliant line, the line 486 $\mu\mu$ as a bright one. No air-lines appear. The apparatus used by the authors, which they believe to be the first simple device for producing Lecoq de Boisbaudran's arc, is made by Franz Hugershoff, Leipzig.

G. C. J.

Detection of Nitrous Acid in the Presence of Nitric Acid. A. Leclère. (*J. Pharm. Chim.*, 1913, **8**, 299.)—The test is based upon the fact that citric acid liberates nitrous acid from its salts, but does not affect nitrates. The solution under examination is treated with an equal volume of a syrupy solution of citric acid, and a little of a 3 to 4 per cent. solution of ferrous ammonium sulphate pipetted on to the surface of the liquid. The characteristic brown ring is obtained in the presence of 1 part of nitrous acid per 10,000, whilst concentrated solutions of nitrates give no coloration in twenty-four hours. A preliminary treatment with zinc acetate, followed by filtration, is necessary when sulphides are present. C. A. M.

Electrometric Titration of Dichromate Ion with Ferrous Iron. G. S. Forbes and E. P. Bartlett. (*J. Amer. Chem. Soc.*, 1913, **35**, 1527-1538.)—Certain reducing agents increase the oxidising potential of the dichromate ion on platinum by amounts up to 0·2 volt. No other oxidising agent tried exhibits a similar effect. The potential increases up to the very endpoint of the reaction, and is highest when the dichromate concentration is least. A final drop of $\frac{N}{10}$ reducing agent often depresses the potential by 0·5 volt. These observations have suggested an improvement in the electrometric method of titrating dichromate and ferrous salts which makes it both more rapid and more accurate than the ferricyanide method.

The solution to be titrated is placed in *B*. The platinum electrode *E* is sealed to the glass tube *T*, which is preferably arranged to be rotated mechanically by a pulley, *P*. Connection is made between *E* and *L* by filling *T* with mercury. *C* is a calomel electrode containing mercury, mercurous sulphate, and potassium chloride. The plunger *F* serves to sweep out the syphon *N* just before the end of a titration. *QR* is a thin wire stretched over the wood-strip *W*. The connection to the battery *V* is made by a piece of wire of such length that the sliding contact *S* is near *R* at the start of a titration. *G* is a mirror galvanometer and *M* its mirror. To titrate a dichromate solution with ferrous salt, the galvanometer is first brought to rest by moving *S*, then ferrous salt is run in, and *S* moved towards *Q* sufficiently to neutralise the anomalous increase in potential. If ferrous salt is to be titrated, a slight excess of dichromate is added, and the mixture titrated back with ferrous solution of known strength. Very soon a nearly constant maximum potential is reached, so that the operator can abandon the sliding contact and occupy his hands solely with the burette and his eyes in watching the spot of light reflected by the galvanometer. Finally, a single drop of $\frac{N}{10}$ ferrous solution in excess drives the spot of light right off the scale. This last drop is subtracted from the volume used. The improvement claimed for this method over those previously described lies in the constancy of the E.M.F. right up to the endpoint, and the simplicity of apparatus and method which can be employed in consequence.



Taking the results obtained by this method as establishing the true endpoint in titrations of ferrous ion by means of dichromate, it is shown that the disappearance of the ferricyanide reaction, when used as a spot-test in the usual way, does not indicate absence of ferrous iron, but a reduction of the concentration of ferrous iron to 0·0003 grm. per 100 c.c. G. C. J.

Iodimetric Estimation of Iron. **C. Beck.** (*Chem. Zeit.*, 1913, **37**, 1330-1331.)—The reaction between ferric salts and hydrogen iodide does not proceed quantitatively unless one of the products of the reaction is removed continuously. If the resulting iodine is removed by distillation, however, the reaction proceeds to completion, and the iron may be estimated with an error not exceeding 0·2 per cent. by titrating the iodine with thiosulphate. The author introduces the ferric solution into an appropriate distillation apparatus, and adds, in the following order, some broken pot, 2 grms. potassium iodide, 1·5 grms. marble in small pieces, 40 to 50 c.c. water, and 20 c.c. concentrated hydrochloric acid. The apparatus is promptly closed, its contents are distilled nearly to dryness, and the iodine is received in a solution of potassium iodide and titrated with thiosulphate. G. C. J.

Determination of Phosphorus in Vanadium Steel and Ferrovanadium. C. F. Sidenerand P. M. Skartvedt. (*J. Ind. and Eng. Chem.*, 1913, **5**, 838-839.)—The method depends on the separation of phosphorus from vanadium by precipitation as aluminium phosphate under closely defined conditions. The sample is dissolved in dilute nitric acid, aided, if necessary, by a little hydrochloric acid. The solution is evaporated to dryness, and the residue is baked to decompose nitrate of iron, and then dissolved in concentrated hydrochloric acid, to which about 0·02 grm. aluminium is added in the form of chloride. The solution is nearly neutralised with ammonia, heated nearly to boiling, and the iron reduced by addition of ammonium bisulphite solution. When reduction is complete, ammonia is added until a slight permanent precipitate of ferrous hydroxide results. This is redissolved by addition of a few drops of ammonium bisulphite solution, and 1 to 2 c.c. of phenylhydrazine is next added drop by drop with constant stirring. If no precipitate appears, ammonia is added drop by drop until one forms, and this is followed by a few drops more of phenylhydrazine. The mixture is boiled for two minutes, and the precipitate allowed to subside and then filtered off. It consists of aluminium phosphate and hydroxide, more or less contaminated with vanadium. It is washed with hot water until the washings develop no cloudiness with mercuric chloride solution, and is then dissolved in dilute nitric acid, the solution being caught in the original beaker. The vanadium is oxidised by a little hydrogen peroxide, and a slight excess of sodium carbonate is then added. The solution is boiled for five minutes, and the phosphorus re-precipitated as aluminium phosphate by gradually adding dilute nitric acid until the solution no longer gives an immediate brown tinge to turmeric-paper. The precipitate is filtered off and washed with 1 per cent. ammonium nitrate. It is then dissolved off the filter with dilute nitric acid and tested for vanadium with hydrogen peroxide. If the original steel contained less than 1 per cent of vanadium, no trace of colour will develop, showing absence of vanadium, and the precipitation of the phosphorus as molybdate may be proceeded with. With more than 1 per cent. of vanadium present, a pinkish-yellow colour may develop, whilst with 5 per cent. present a deep red colour will probably appear. In such cases a second precipitation with sodium carbonate and nitric acid is necessary, and ferrovanadium with high percentages of vanadium may call for a third precipitation. An ordinary vanadium steel with 0·15 per cent. vanadium and (according to the U.S. Bureau of Standards) 0·035 per cent. phosphorus was found to contain 0·0340 and 0·0356 as the extreme values of a series of analyses. With high-grade ferrovanadium (30 per cent.) the method may give results nearly 10 per cent. in error, but on such material the exact percentage of phosphorus is less important and its determination difficult by any method. G. C. J.

Colorimetric Method for Titanium in Iron and Steel. C. R. McCabe. (*J. Ind. and Eng. Chem.*, 1913, **5**, 735-736.)—The method described is the ordinary peroxide method, with extraordinary precision in compensating for the colour of ferric salts, which, in the case of titanium steel, may be more intense than that due to titanium. The sample (2 grms.) and an equal weight of titanium-free steel are dissolved in separate flasks by heating with 80 c.c. of dilute (1 : 3) sulphuric acid. Nitric acid (2 c.c.) is added to the contents of each flask, and boiling continued until the solutions are free from fumes. The cooled solutions are washed into similar Nessler cylinders, filtering the test solution through a plug of cotton if the sample under examination is cast iron. The colours will either match, or that of the standard steel, selected for its high content of iron, may be slightly more intense. In this case a little of it is rejected until the solutions viewed from above appear identical in tint. Hydrogen peroxide (2 c.c. of a 3 per cent. solution) is added to the contents of each cylinder, when a distinct difference in tint will be noticed if the sample under examination contains 0·02 per cent. of titanium. The tints are again equalised by adding a standard solution of titanium to the control solution in the usual manner, taking care, unless the amount of titanium be very small, to secure equality of volume between the comparison liquids before making the final comparison.

When less than 0·02 per cent. of titanium has to be looked for and estimated if found, it is best to make an ether separation of the greater part of the iron. The sample and control steel are dissolved in 50 c.c. hydrochloric acid, and each solution is oxidised by means of nitric acid and evaporated to about 10 c.c. It is then washed into a separator with about 15 c.c. hydrochloric acid (2 : 1), and extracted with 50 c.c. ether. The acid layer is run off into a beaker, making the separation with care that not more than a drop of the ethereal solution passes into the beaker in either experiment. The solution is diluted with 200 c.c. hot water, made alkaline with ammonia, boiled, and filtered. The precipitate is washed free from chlorides and dissolved by placing the filter in a dish and treating it with 10 c.c. dilute (1 : 3) sulphuric acid. The solutions, which have a light maroon colour, due to a little iron, are filtered into comparison tubes, and the analysis completed as usual. Hydrogen peroxide discharges the maroon colour of the control solution, which gives place to a very pale green. The test solution, in presence of titanium, develops the colour characteristic of that metal, of which as little as 0·001 per cent. can readily be detected. When using this refined method to discover the ultimate distribution of very small quantities of titanium, which, though used in course of manufacture, do not necessarily appear in the finished iron or steel, the insoluble matter from cast iron must be ashed, freed from silica, fused with sodium carbonate, and the acid solution of the product of fusion added to the main solution. The insoluble matter from cast iron always contains titanium, if titanium is present in the iron, but in ordinary technical work the amount may be neglected.

The paper describes a method for the preparation in bulk of standard titanium solution with commercial ferrotitanium as starting-point. G. C. J.

Estimation of Vanadium in Steel by the Hydrogen Peroxide Colour Method. C. R. McCabe.

(*J. Ind. and Eng. Chem.*, 1913, **5**, 736-738.)—For the determination of vanadium, even in the difficult case where this metal is accompanied by titanium and molybdenum, the author has already described a method (ANALYST, 1911, **36**, 473). This method is still recommended in such cases, and in the equally troublesome case of a steel containing a small amount of vanadium with much chromium. In most cases, however, satisfactory results can be obtained by simpler means. In absence of chromium, 2 grms. of the vanadium steel and an equal weight of a non-vanadium steel are dissolved separately in 40 c.c. dilute (1 : 1) nitric acid. About 0·1 grm. permanganate is added, and the mixtures are digested for two minutes. The solutions are then clarified by addition of ammonium bisulphite, and boiled to expel sulphur dioxide. They are next cooled, made equal in volume, and their colours, which should be identical, compared. Into each tube 1 c.c. of 3 per cent. hydrogen peroxide is added, and, to the control solution, a standard solution of vanadic acid until the colours match, adding water to the assay from time to time to keep the volumes approximately equal.

In presence of chromium, 2 grms. of the vanadium steel is dissolved in 80 c.c. of dilute (1 : 3) sulphuric acid, and 2 grms. of steel free from vanadium or chromium is dissolved in an equal quantity of acid, to which potassium bichromate, equivalent to the chromium in the assay, has been added. When nearly dissolved, 25 c.c. of concentrated nitric acid is added, and the mixture heated for ten minutes. Vanadium is not completely oxidised by a small amount of nitric acid such as would oxidise the iron. The analysis is then finished as in absence of chromium. The method overcomes difficulties which arise from the fact that the colour and intensity of colour of chromium compounds vary with the acidity of the solution and other factors not readily determined. G. C. J.

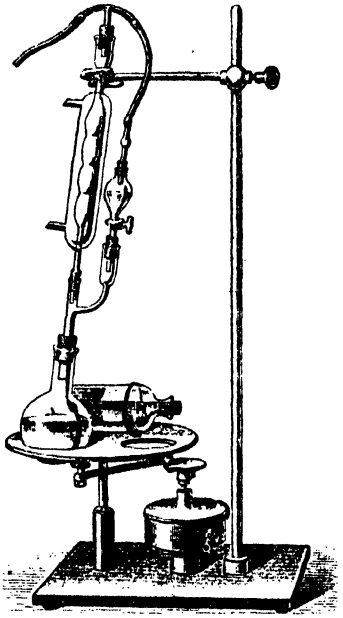
Estimation of Zinc in Treated Ties (Railway Sleepers). F. C. Frary and M. G. Mastin.

(*J. Ind. and Eng. Chem.*, 1913, **5**, 738-739.)—Railway sleepers, treated with zinc chloride, are usually sold to a specification of 8-oz. zinc chloride per cubic foot, or 0·5 per cent. metallic zinc. Except with oak, the estimation of zinc may be accomplished by charring borings with sulphuric acid, adding nitric acid a few drops at a time, continuing the heating until all organic matter is destroyed and nitrous fumes driven off, dilution, removal of iron, precipitation of zinc as sulphide, and final weighing as oxide.

With oak sleepers, especially red oak sleepers, the above method fails, for the treatment with acid, no matter how prolonged, does not destroy all organic matter, and the precipitation of the zinc in the dark coloured solution is so incomplete that the estimation may be 50 per cent. in error. All kinds of wood yield to the following treatment: The borings (2 grms.) are weighed into a 200 to 300 c.c. nickel dish, moistened with water, and warmed to insure saturation. About 30 grms. of potassium hydroxide is added, and the mixture heated to fusion. Whilst stirring with a nickel wire, the heat is increased until the mixture boils vigorously. Lumps of gummy substance, which separate, gradually disappear, and the fusion begins to foam. When spurts of inflammable gases appear, about 0·1 grm. of potassium

nitrate is added, and similar additions, perhaps five in all, made, with constant stirring, until the fusion becomes transparent, colourless, and tranquil. The cooled mass is dissolved in water, 15 to 20 c.c. bromine water added, and the mixture boiled for a minute. Nickel hydroxide, thus precipitated, is filtered off on a Gooch crucible, and the filtrate and washings transferred to a large (750 c.c.) dish, and made faintly acid with concentrated hydrochloric acid. Excess of sodium sulphite is added—a little at a time, until the first violent effervescence ceases—and then 10 to 20 c.c. of concentrated hydrochloric acid. The solution is diluted to 400 c.c., heated to boiling, and titrated with ferrocyanide, using a saturated solution of uranium nitrate as outside indicator, or zinc may be determined by any other method. Enough sulphite must be used to reduce all oxidising compounds, which might otherwise decompose ferrocyanide, and the latter must of course be standardised under approximately identical conditions as regards dilution, acidity, and saline concentration.
G. C. J.

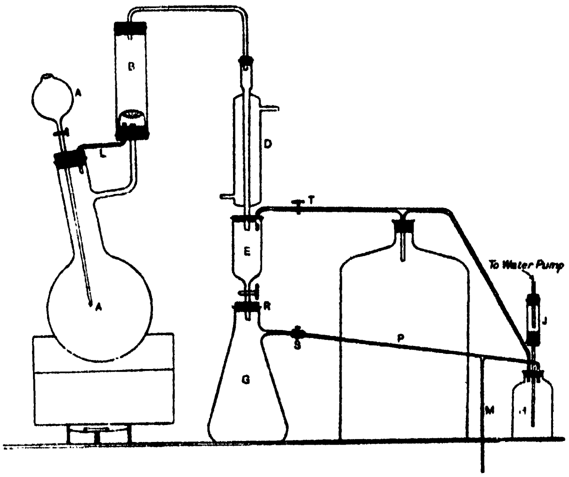
APPARATUS, ETC.



Mechanical Agitator. V. Grignard. (*Bull. Soc. Chim.*, 1913, **13**, 952-955.)—The apparatus here shown will be found useful for the agitation of flasks while connected with a reflux condenser. It consists essentially of a circular iron or copper plate, which revolves horizontally on a vertical axis. A slight oscillating movement (about 5 to 6 cm.) is communicated to it by means of a crank beneath, which is connected with an ordinary laboratory turbine. The upper part of the reflux condenser is supported in a ring of wide diameter. The surface of the plate is covered with asbestos, and is pierced with openings to receive the flask, which may thus be made to oscillate over a flame or an electric lamp. The apparatus may be obtained from Lequeux, 64, Rue Gay-Lussac, Paris.
C. A. M.

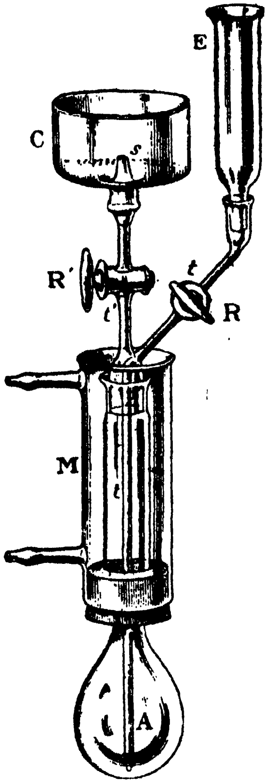
Bomb with Stirring Gear for the Measurement of Reaction-Velocity in a Heterogeneous System at High Pressures. New High-Pressure Reducing Valve. L. Stuckert and M. Enderli. (*Chem. Zeit.*, 1913, **37**, 1288.)—The bomb consists of an upright Mannesmann tube, closed at each end by a conical plug forced into its seat by a threaded cap. The liquid and gas are admitted or removed through capillary copper tubes passing through glands. All joints are made tight by the method of F. Haber and R. Le Rossignol (*Zeitsch. Elektrochem.*, 1913, **19**, 57). In the axis of the upper plug a sleeve is fitted gas-tight by means of a reversed conical seating; the axial bore of this sleeve is enlarged into two oil-chambers, and carries a nicely fitting steel shaft, the upward thrust of which is taken on a ball thrust-bearing. Absolute tightness is secured by a slightly conical Klingerite washer at each end of the sleeve. Side-pull from the driving-band is prevented by outboard bearings above and below the pulley. A double-walled oil-intercepting cup and a Witt stirrer are carried at the lower end of the shaft. The heating with pressures up to 80 atmospheres at 2,000 revolutions per minute for runs of five to ten hours was so small that kinetic determinations at constant temperature could be carried out. The new valve is constructed on the principle of the Haber and Le Rossignol joint. It gives a large opening for a small movement of the spindle; its packing consists of easily renewed vulcanised fibre disks, and it is not subject to erosion of the valve seat. O. E. M.

Apparatus for the Continuous Evaporation of Large Volumes of Liquid under Diminished Pressure. W. A. Davis. (*J. Agric. Sci.*, 1913, **5**, 434.)—The apparatus was specially devised for dealing with alcoholic plant extracts which have a tendency to froth, and it can be left without attention when once started, so long as the liquid in the distilling flask *A* is renewed from time to time from the dropping funnel A^1. *B* serves as a froth trap, the copper gauze seen at the base of it serving to break up the froth and insure its return through the side tube *L* to the distilling flask *A*. The large reservoir *P* takes up small variations in the water pressure, and so insures a steady vacuum, which is maintained by an ordinary water injector pump connected through a Hutchinson regulating valve, *J* (Analyst, 1912, **37**, 480), with the bottle *H*, and thence with *E* and *G*. The combination *E* and *G* allows of the distillate being removed at any time, the vacuum being meanwhile maintained by closing the cock on *E* and the screw clamp *S*. *G* can then be emptied and replaced, and by first opening *S* alone the vacuum in *G* is soon re-established. H. F. E. H.

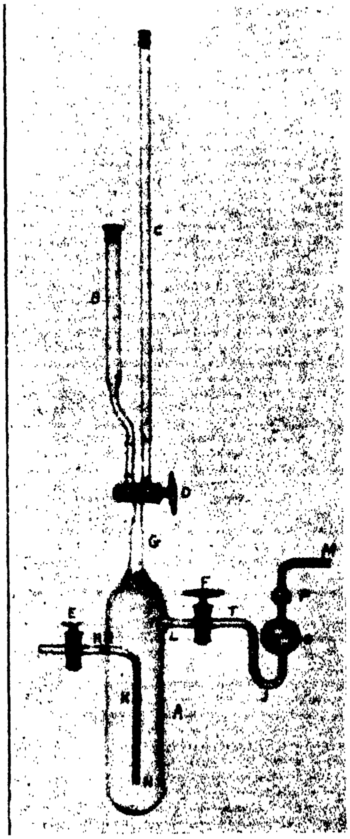


Apparatus for Determining the Density and Internal Friction of Gases. M. Hofsäss. (*J. Gasbeleuchtung*, 1913, **56**, 841; through *Chem. Zentralbl.*, 1913, II., 1353.)—The apparatus, arranged as a bye-pass in the gas-supply, consists essentially of a chamber provided with a manometer and a fine jet controlled by a cock, and closed at the ends by stopcocks. The gas, collected in it under pressure, is allowed to flow out through the jet; the time required for the manometer liquid to fall from one mark to another is observed by a stop-watch, and affords a measure of the density, which is proportional to the square of the time of flow. If the service-pressure is insufficient, gas enclosed in a second chamber may be forced into the first by a levelling vessel. To determine the internal friction, a capillary is substituted for the jet. O. E. M.

Apparatus for Extracting Carbon Monoxide from Blood. M. Nicloux. (*Bull. Soc. Chim.*, 1913, **13**, 947-952.)—The use of the apparatus shown in the figure obviates the necessity of a mercury pump for extracting carbon monoxide from blood. The flask A, which has a capacity of 40 to 200 c.c., according to the quantity of blood available, is charged with phosphoric acid (40 c.c., or one and a half times the volume of the blood) and 25 c.c. (or the volume of blood) of water, and is connected with the refrigerator M. The tube t is filled with water, the tap R closed, and the tap R' opened. The flask is then three parts immersed in a calcium chloride bath at 110° C., and a vacuum created by means of a water-pump attached to S, after which the tap R' is closed, and water is introduced into the upper part of the tube t'. The blood (25 c.c.) is now introduced into E, and allowed to fall very slowly into the flask through the tap R, the tubes E and t being subsequently washed with 15 c.c. of water. In contact with the phosphoric acid the blood parts with its carbon monoxide, whilst a part of the pigment and proteins is dissolved. To remove froth from the neck of the flask, the apparatus is withdrawn from the bath, cooled with a jet of water, and 10 to 15 c.c. of hot water are introduced from the basin C by rapidly turning the tap R'. If necessary, this operation is repeated two or three times. After fifteen to twenty minutes the extraction of the carbon monoxide will be complete, and the flask is withdrawn from the bath. Hot water is now admitted through the tap R until there is no longer a vacuum. The basin C is then filled with water, a measuring cylinder placed over S, the tap R' opened, and the gas collected and measured. In addition to its use for the examination of blood in cases of carbon monoxide poisoning, this apparatus may be used for the collection of any gas insoluble in water generated by the interaction of two substances. C. A. M.

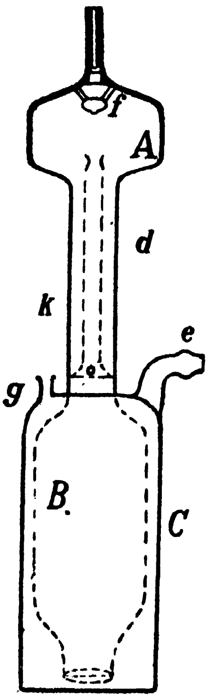


Simple Gasoline Gas Generator for Sulphur Determinations. C. E. Waters. (*J. Ind. and Eng. Chem.*, 1913, **5**, 853-854.)—The apparatus consists of a glass tube 25 cm. high and nearly 5 cm. in diameter, closed at both ends, but with a tube sealed through the top and extending nearly to the bottom, and another tube sealed on to the side near the top. The tube is charged for a third of its height with glass beads, and for another third with petroleum spirit, or a mixture of this with gasoline; gasoline alone is unsatisfactory. The apparatus is supported in a suitable stand, and the long glass tube which extends to the bottom of the layer of beads is connected to a supply of compressed air at a pressure of about 40 inches of water, and the side tube is connected to a gauze-topped safety burner. Black rubber tubing is used for the connections. The object of the beads is to break the stream of air up into small bubbles, without which it would be impossible to obtain a steady flame. The size of flame is easily regulated by a cock on the burner or clip on the tube near it, the supply of compressed air behind the generator being always full on. The apparatus has been in use for several years without accident in the laboratory of the U.S. Bureau of Standards, the only precaution necessary being to extinguish the flame before turning off the supply of compressed air. G. C. J.



Apparatus and Method for Determining Hydrogen Sulphide in Illuminating Gas. E. P. Harding and E. Johnson. (*J. Ind. and Eng. Chem.*, 1913, **5**, 836-838.)—The 300 c.c. bulb, *A*, of the apparatus figured is charged with 100 to 150 c.c. of a strong solution of cadmium chloride, of which 5 to 6 c.c. are tilted over into the bulb attachment *J*. The stopcock, *D*, being closed, and *E* and *F* open, *M* is attached to a meter and *E* to the supply of gas to be tested, the speed of which is adjusted to 1 to 1·5 c.ft. per hour. If the liquid in *O* develops a coloration, the gas is cut off and the meter read. In testing partially purified gas, 0·1 c.ft. is a convenient quantity to take; but if no precipitate is visible in *A* when 0·1 c.ft. has passed, the experiment is continued until one appears. The final reading of the meter having been taken, the apparatus is disconnected from the meter and gas-supply, *D* is opened, and the liquid in *O* tilted into *A*. Wash-water is then drawn into *A* through *M* by applying suction at *B*, and more wash-water is similarly sucked in through *E*. The gas above the solution in *A* is next replaced by air by applying suction at *B* with *F* open; some starch solution is run into *A* through *B*, and the air in *A* is placed under diminished pressure by suction applied at *B*. Concentrated hydrochloric acid is run into *A* through *B* until the precipitate of cadmium sulphide is dissolved. A small excess of acid is allowed to run in, and the liberated hydrogen sulphide is finally titrated with a weak solution of iodine delivered from *C*. For unpurified coal-gas $\frac{N}{30}$ is a convenient strength for the iodine solution, a solution one-tenth as strong serving well for purified gas; or the strength may be adjusted so that each c.c. corresponds to 10 grains or 1 grain of hydrogen sulphide per 100 c.ft., when 0·1 c.ft. is taken for the test. The results are about

5 per cent. lower than those obtained by the gravimetric cadmium chloride method, which is admitted to be more accurate. On the other hand, the gravimetric method is slow, and includes sulphur compounds other than hydrogen sulphide. G. C. J.



Gas Analysis Apparatus. A. Lomschakow. (*Chem. Zeit.*, 1913, **37**, 1262.)—An absorption vessel for use with volumetric gas analysis apparatus is illustrated in the figure. It consists of an upper working vessel, *A*, which is connected by a capillary tube with the measuring burette, and two lower vessels, *B* and *C*, which contain the reagent. The inner vessel, *B*, is open below, and thus in communication with the outer vessel, *C*; it is also connected with the working chamber, *A*, by means of the central tube, *d*. The reagent is introduced through the tubulure, *g*, which is closed by a rubber stopper. To the side-tube, *e*, a rubber balloon is fixed and filled with air. On gradually compressing the balloon the reagent is forced from *C*, through *B* and the central tube, *d*, into the absorption chamber, *A*, and when the latter is full the capillary tap is closed, and the apparatus is ready for use. When in use, the gas is admitted into the vessel *A*, displacing the reagent, which flows at first down the tube, *d*, and later through two small holes situated at the bottom of this tube, through which the gas bubbles up. Absorption begins at once, and is continued by closing the capillary tap leading to the burette and squeezing the rubber balloon sharply. This has the effect of projecting a spray of the reagent up the central tube, *d*, against the baffle-plate, *f*. The liquid flows back again down the walls of the vessel, and in passing back into the chamber *B*, through the small holes at the bottom of the tube, *d*, it comes into intimate contact with the displaced gas. After repeating the operation a few times absorption is complete, as may be confirmed by means of graduations on the outer tube, *k*; the gas is then returned to the burette. This type of absorption vessel may be fitted to Orsat's apparatus; it has the advantage of holding double the quantity of reagent contained by the ordinary vessel; the whole quantity of the reagent takes part in the absorption, and the danger of some of the liquid being driven over into the measuring-tube is minimised. J. F. B.

Use of the Sense of Smell in Volumetric Estimations. J. F. Sacher. (*Chem. Zeit.*, 1913, **37**, 1222.)—Certain substances possessing a distinct odour, the neutral salts of which are odourless, may be titrated without the use of an indicator, the disappearance of the odour being taken as the end-point of the titration. The author has observed, however, that the appearance of an odour can be more readily detected than its disappearance, and the substance should therefore be treated with a slight excess of the neutralising reagent, and this excess titrated back again. Approximately correct results may be obtained in the case of valerianic acid, acetic acid, and ammonia; but in many cases, especially that of the higher phenols, the hydrolytic action of atmospheric carbon dioxide renders the method useless. W. P. S.

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LAW REPORT.

Demerara Sugar: Anderson v. Britcher. (High Court of Justice, King's Bench Division, Divisional Court, November 12; before Mr. Justice Darling, Mr. Justice Avory, and Mr. Justice Atkin.)—This was a special case stated by a Metropolitan Police magistrate sitting at Clerkenwell Police Court. Mr. Barrington-Ward appeared for the appellant, and Mr. G. Elliott, K.C., and Mr. Guy Lushington appeared for the respondent.

On July 23 last an information was laid by the appellant against the respondent under Section 6 of the Sale of Food and Drugs Act, 1875, for that on June 30 he unlawfully sold to the prejudice of the purchaser a certain article of food—to wit, "Demerara sugar," which was not genuine Demerara sugar, but cane-sugar crystals coloured with an organic dye foreign to genuine Demerara sugar, so that the sugar was not of the nature, substance, and quality demanded.

The magistrate dismissed the information.

The following facts were proved or admitted: The respondent was a grocer carrying on business at King's Cross Road. On June 30 the appellant called at the shop and asked the respondent for 1 pound of Demerara sugar, and received from him 1 pound of sugar, for which he paid 2½d. The appellant had it analysed, and the analysis showed that the sugar was cane-sugar crystals coloured with an organic dye foreign to genuine Demerara sugar. It was, in fact, a crystallised cane-sugar grown in Mauritius. Sugar grown in Demerara and in other West Indian Islands is a crystallised cane-sugar. Originally it is white, but it is treated so as to become yellow. The colour has been varied from time to time to suit the taste of the public. The colour was formerly obtained by the action of chloride of tin, which converted portions of the sugar into caramel; but that process left traces of tin in the sugar, and a later process was treatment with a colouring matter known as "golden bloom," which contains an organic dye. Large quantities of that colouring matter are exported from England for use in Demerara and the West Indian Islands. The sugar in this case was coloured by the modern process with an organic dye, the colouring matter being infinitesimal in amount and quite harmless. Before the magistrate evidence was given that the sugar was equal to the best grade of West Indian cane-sugar, and that the public expect under the designation of Demerara sugar a yellow crystallised cane-sugar without reference to its place of origin. The appellant admitted that the term "Demerara sugar" might be properly applied to similar sugar produced in Jamaica and other West Indian Islands, but not elsewhere. No disclosure was made to the appellant at the time of purchase that the sugar, though sold as Demerara, was not in fact produced in Demerara or in the West Indian Islands.

The appellant contended that the name implied that the sugar had been produced in Demerara, and that as no disclosure had been made that it was not so produced, an offence had been committed. He contended also that the nature and quality of Demerara sugar depended on the peculiar climate and soil of the particular district, so that, though the description might be applied to sugar produced in the West Indian Islands as well as in Demerara, it could not properly be applied to sugar produced elsewhere.

For the respondent it was contended that the Act aimed at securing that the article purchased should be of the nature, substance, and quality demanded, and that the place of origin was immaterial. And it was further contended that "Demerara sugar" had become a generic term, referring to a process of manufacture, and not to a place of production; and the magistrate accepted these contentions.

Mr. BARRINGTON-WARD now pointed out that there was no question under the Merchandise Marks Act, and he had to show an offence under the Sale of Food and Drugs Act, 1875. The magistrate had so stated the case that he could not say the article was defective in either substance or quality, but he submitted that it was defective in nature. The point under the Act was not what the purchaser expected to receive, but what the seller was bound to supply. The true meaning of "nature" included a reference to place of origin.

Mr. ELLIOTT pointed out that a Bath bun or a Cambridge sausage was not an article produced only at Bath or at Cambridge.

JUDGMENT.

Mr. JUSTICE DARLING said that the appeal must fail. The magistrate had so stated the facts that argument was hopeless. Demerara sugar had become a conventional term, and this sugar was Demerara sugar in every single respect except that it was not grown in Demerara. Indeed, if a purchaser asking for Demerara sugar were offered genuine sugar grown in Demerara in its natural state and colour, he would probably refuse it. No one supposed that a Brussels carpet was necessarily made in Brussels, and Demerara sugar might be so without being grown in Demerara.

Mr. JUSTICE AVORY concurred, but said that he wished to guard against being taken to say that sugar grown anywhere might be called Demerara merely because it was cane-sugar and coloured yellow.

Mr. JUSTICE ATKIN concurred.

Solicitors: Messrs. W. T. Ricketts and Son; Messrs. Neve, Beck, and Kirby.—*Times Financial Supplement*, November 13, 1913.

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REVIEWS.

ALLEN'S COMMERCIAL ORGANIC ANALYSIS. By DAVIS and SADTLER. Vol. VII. London: J. and A. Churchill, 1913. Price 21s. net.

In this volume the article on Vegetable Alkaloids has been written by G. Barger, Glucosides by E. F. Armstrong, Animal Bases by A. E. Taylor, Ptomaines or Putrefaction Bases by G. Barger, Animal Acids by J. A. Mandel, and Lactic Acid by W. A. Davis.

Cyanogen and its Derivatives, by Herbert Philipp.—In the preparation of an article on the commercial analysis of any group of substances, the compiler has a far from easy task. Not only is he confronted by the usual difficulty of the selection of the most suitable methods from a very large number, but there is the further disadvantage that many of the methods in use are published in special journals which may not be readily available for reference, whilst others regularly in use for commercial analysis, and modifications of methods whereby increased rapidity and accuracy are obtained, are frequently not published at all. After making every allowance for these obstacles, however, it must be confessed that the article on cyanogen and its derivatives in the new "Allen" is, in many respects, very disappointing.

Of the 106 pages covered, over a third are taken up with a textbook description of the properties of a large number of cyanogen derivatives and references to their methods of manufacture. Whilst it is doubtless advantageous to mention the properties and methods of manufacture of the occurring commercial salts, so far as these have a bearing on the methods of analysis, it seems to be a waste of valuable space in a work such as this to describe derivatives not occurring in commerce, which information any analyst can look up equally well in the usual books of reference. Further, whilst the statements made as to properties are generally correct, a number of errors occur. Thus, for example, Conroy's table of the solubility of sodium ferrocyanide is reproduced on p. 506, but the decimal point has been misplaced throughout, so that the solubilities given are only one-tenth of the correct figures. Again, it is stated that the ferrocyanides of magnesium and the alkali earths are "somewhat" soluble in water, which is only true of the barium salt, those of magnesium, calcium and strontium being exceedingly soluble in water, more so than those of the alkali metals. The paragraph on the constitution of the various "Prussian blues" remains as in the last edition, no reference being made to the investigations made since then by K. A. Hofmann and others. Where any information is given as to methods of manufacture, it is for the most part very unsatisfactory, as, except for one or two slight additions, such information remains the same as in the 1896 edition of "Allen," although during the past eighteen years the whole industry of the cyanide, ferrocyanide, and sulphocyanide manufacture has undergone a complete change.

So far as analysis proper is concerned, the most satisfactory part of the article is that relating to the analysis of the simple cyanides, in which a good account is given of the methods of analysis of the commercial products for their cyanogen content and the commonly occurring impurities, and also for the analysis of cyanide solutions in gold works for the control of the extraction process, the methods for the latter purpose given by Clennel in "The Cyanide Handbook" being fully described.

That part of the article dealing with the commercial analysis of ferrocyanides is most disappointing, and the same is true to a lesser extent in the case of the thiocyanates, and here it cannot be said that the statement in the General Preface to the new edition is complied with. It is there stated that "obsolete methods are omitted; what little of the old text remains has been carefully revised." In fact, most of the methods mentioned in the 1896 edition of Allen, at a time when comparatively little had been done on the subject, are reproduced in the present edition, including some errors occurring in the former book. Some new methods have been added, which in many cases appear to have been copied from an abstract, and not from the original paper. A number of the methods given are quite obsolete, and others, though still in use, have been considerably modified to increase their speed and accuracy. In the case of the Knublauch method for the estimation of ferrocyanide by titration with copper sulphate solution, which is largely used for commercial purposes, the author gives a full account of the precautions required so far as these were given by Knublauch in 1892, but makes no mention of the further precautions which later experience has also shown to be necessary. In addition, although mention is made of the existence of carbonylferrocyanides, and a description of their properties is given, nothing is said as to their presence in the ferrocyanide products obtained from crude coal-gas, and of the influence which these have in analysing such products.

So far, therefore, as the commercial analysis of ferrocyanides and thiocyanates is concerned, it must be concluded regretfully that the article is not likely to be of much use to analysts who are dealing with these products, and still less to those unacquainted with the special subject who are desirous of obtaining information on the matter. HAROLD G. COLMAN.

Non-Glucosidal Bitter Principles, by G. C. Jones.—The name of Mr. G. C. Jones at the head of this section, a considerable part of which is concerned with the chemistry of hops, is a sufficient guarantee that it has been written with a discriminating and well-informed pen. The somewhat indefinite character of certain of the bitter principles in question and the voluminous and confused literature in which their properties are described make it a matter of great difficulty to distinguish between the true and the false, and between what is really useful to the analyst and what is, at best, of merely academic interest. To say that Mr. Jones has succeeded well is to pay him a high compliment.

Since this volume was published, or perhaps while it was in the press, several papers dealing with the chemistry of hops have appeared, and consequently a few of the statements stand already in need of revision. The statement at the top of p. 165, for example, could hardly be made with the same assurance since the publication of Professor A. J. Brown's recent paper. Brown has shown (what others have long suspected) that the preservative properties of hops and the percentage of so-called "soft resin" do not stand in any direct proportionality, and that a period of extraction quite insufficient for the solution or removal of the resins will suffice to extract the substance or substances on which the preservative properties of hops depend. Notwithstanding this, there can be no doubt that the estimation of the "soft resins" is of considerable importance, and that it still affords one of the best criteria of the brewing value of a sample of hops.

The origin and nature of the γ-resin is of considerable interest, but, as Mr. Jones points out, it is certainly not derived from the essential oil. Power has recently shown that the resinous constituents of hops include a considerable amount of fatty acids, and on this ground has cast a good deal of doubt on the accuracy of the results obtained by Lintner's titration method. We must, however, suspend judgment on this point for the present, since there are still very many points in connection with the complex and elusive substances constituting the resins of hops which must be cleared up before the final word is spoken.

With regard to the estimation of arsenic in hops, the reviewer's experience has been that, as a rule, higher results are obtained when working directly on the hops than when the organic matter is destroyed by the ignition method described. With Mr. Jones's conclusions as to the significance of arsenic in hops and what may be called the permissible "advisory" limit, the reviewer is in complete agreement.

The detection of hop substitutes in beer is a matter of little or no practical importance, and, recognising this fact, Mr. Jones has wisely condensed this portion of the subject into a couple of pages, which is in reality more than sufficient.

In conclusion, the reviewer is of opinion that this section of the seventh volume will prove a trustworthy guide to any analyst who, not being a specialist, is desirous of obtaining reliable information as to the analytical chemistry of the non-glucosidal bitter substances with which it deals. A. CHASTON CHAPMAN.

THE APPLICATION OF PHYSICO-CHEMICAL THEORY. By R. KREMANN. Translated by H. E. POTTS. Constable and Co. 1913. Price 8s. 6d. net.

The present volume is one of a series of monographs on the manufacturing methods of technical chemistry, and this fact bears witness to the increasing importance of physico-chemical theory for the rational control of laboratory and factory processes. In the nature of things physical chemistry has been, and still is, very largely a theoretical branch of the science, but in its later developments it has coordinated in a remarkable manner the empirical observations of technology, and formulated definitely the conditions under which alone various technical operations can be successfully carried out. It is true that the majority of these processes to which physico-chemical theory has been thus applied had already been worked out empirically to a successful issue, but cases can now be quoted where the physical chemist has made a definite contribution to the solution of a technical problem. The synthesis of ammonia is a notable instance of this, but, curiously enough, it is not discussed in the present volume.

An introductory chapter on the two laws of thermodynamics is followed by consideration of reaction velocity and catalysis, the law of mass action, and the phase rule, various technical processes and problems being discussed at the appropriate place. In this way the Deacon process, the drying of linseed oil, the vulcanisation of rubber, the sulphuric acid contact process, vitrification and devitrification, the setting and hardening of cement, the iron-carbon alloys, the reactions of the blast furnace, the manufacture of soap, the ammonia-soda process, the theory of dyeing, and other matters, are treated from the physico-chemical standpoint.

The volume is thus a fairly comprehensive survey of the practical bearings of physical chemistry, and will be found highly suggestive by those who are already to some extent familiar with this branch of the subject. The author cannot be said to cater for beginners, and there is a lack of clearness sometimes, both in expression and in the arrangement of the material. The following definition of adsorption (p. 176) furnishes an instance where this fault may be found: "Adsorption really signifies that when a colloidal precipitate is brought in contact with a solution, it takes up a certain part of it. The compound so produced is called an 'adsorption compound.'" Further, there might have been incorporated with advantage more of the actual experimental data which show the applicability of general formulæ to particular cases. From the standpoint of the average reader, such data contribute not a little to the interest and clearness of the treatment. J. C. PHILIP.

LIQUID AIR, OXYGEN, NITROGEN. By GEORGES CLAUDE. Translated by HENRY E. P. COTTRELL. With a Preface by D'ARSONVAL. Pp. xxv+418. J. and A. Churchill. Price 18s. net.

Until the appearance of this work, no very recent book had appeared in England dealing with the liquefaction of gases, and the commercial production of oxygen and nitrogen had been left almost untouched. Referring to the growing use of these elements, the translator remarks in his foreword that "this reason, even if no other existed, would have afforded a sufficient inducement for providing a standard work on this subject in the English tongue, an acknowledged want which the present translation, it is hoped, will help to satisfy." The reviewer has sometimes speculated as to the utility of translating foreign technical works; in this particular case he is glad that the work has been done, for the book will now be necessarily stocked in this country and thus be generally accessible, which was certainly not the case even a year after the appearance of the original French edition in 1909.

Anyone desiring an up-to-date exposition of the methods by which oxygen and nitrogen are usually prepared commercially would do well to consult either the original or the translation; and if the reader has any sense of humour, he will forgive occasional irrelevances on account of the amusement he will get out of Claude's descriptions of his own mistakes. Perhaps the third person used in the translation is not quite as attractive as the first person of the original, but the translator is to be heartily congratulated on the way in which he has rendered the sense and style of M. Claude's French.

The nineteen chapters of the book fall into four parts, dealing respectively with the Liquefaction of Gases, the Commercial Liquefaction of Air, the Preservation and Properties of Liquefied Air, and the Separation of the Air into its Elements.

The first part deals with the scientific history of liquefaction from the time of Monge and Clouet to the helium cycle of Kammerlingh-Onnes. Not only is actual liquefying apparatus dealt with, but vapour-tension of liquids, heat of vaporisation, the critical point, etc., are explained in an elementary manner.

The second part, which is concerned with the commercial liquefaction of air, begins with a long chapter (IV.), in which the insufficiency of a multiple cycle process on a commercial scale is pointed out, and the reasons why cold is produced during gaseous expansion explained. The necessity of exchangers is dealt with, and the machines of Hampson and Linde described. Whilst these workers relied on the work done during the expansion of a gas against the attraction of the molecules for reduction in temperature, Claude's ambition was to cool the gas by making it do external work which could be utilised in balancing some of the work done in compression. How success was eventually obtained is described later in Chapter VII., probably the most interesting chapter in the book. One realises the difficulties of lubrication at low temperatures, and must admire the idea of using petrol at the temperature of liquid air for purposes which at ordinary temperature usually require a cylinder oil. It is interesting to know that Claude succeeded in 1912 in doing away with lubrication by substituting stamped leathers for the metallic rings of the pistons; apparently the leather receives some preparation before stamping.

The preservation and properties of liquid air are dealt with in a decidedly popular manner in Part III., whilst Part IV. gives an account of the separation of oxygen and nitrogen from the air. As far back as 1892 Parkinson had the idea of separating the constituents by utilising their difference in vapour-pressure, and he had the sound idea of recuperating the cold produced when the liquids evaporated. Linde and Hampson patented the first successful processes; these and other methods based on fractional distillation are described. Recent advance has been more in the direction of fractional condensation, or rather in a combination of the two. The reason is not far to seek—the first drops of liquid which separate on condensing air contain 47 per cent. of oxygen. The production of pure nitrogen by auto-progressive rectification was patented in 1903 almost simultaneously by R. Levy (who was drowned in the *Titanic* disaster) and Linde; the method has become of great importance with the introduction of the cyanamide industry.

The paper and printing are admirable, and the French blocks which have been employed for the illustrations have come out excellently. The work is, however, defective in that, like the original, it contains no index; this might well be added in another edition, and several misprints eliminated at the same time. Misprints are not generally a serious matter, but become annoying when introduced into mathematics—*e.g.*, the integral on p. 132.

The book will doubtless be widely read and much appreciated.

J. T. HEWITT.

THEORIEN DER ORGANISCHEN CHEMIE VON DR. FERDINAND HENRICH. ZUGLEICH ZWEITE AUFLAGE DER NEUEREN THEORETISCHEN ANSCHAUUNGEN AUF DEM GEBIETE DER ORGANISCHEN CHEMIE. Braunschweig: F. Vieweg und Sohn. 1912. Pp. xiv + 401. 13 Abbildungen. Price 10 marks.

This book, as indicated in the subsidiary title, is, under a new name, the second edition of the author's well-known work on "Recent Theories of Organic Chemistry."

The new title affords a better description of the scope of the work than did the old, since in both editions the discussion of the latest theories is introduced by a carefully written historical review of the early development of theoretical organic chemistry. In the fourteen chapters devoted to the more recent theories the author's endeavour has been to discuss as many as possible of these, with the result that some degree of inequality of treatment has crept in, less than justice being done to some highly interesting subjects. However, difficulties of selection always abound in the treatment of a literature so voluminous as that of theoretical organic chemistry, and on the whole a fair picture of the modern position is presented. The author cannot, however, be acquitted of "insularity" of treatment, and has in many cases confined himself much too exclusively to the literature of his own country. What is to be thought of an account of colour and constitution without mention of Armstrong, of fluorescence and constitution without the name of Hewitt, and of discussions on tautomerism, the Walden inversion, and the relations of optical rotation and constitution, with no reference to the work of Lowry, McKenzie and Frankland, and Patterson respectively?

The author has the gift of lucid exposition, and possesses a lively and trenchant style which greatly adds to the pleasure of the reader. A. HARDEN.

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INDEX TO VOLUME XXXIX.

INDEX OF NAMES.

ABDERHALDEN, E., Utility of the Dialysis Method in Clinical and Biological Problems, 223
Adams, H. M., Commercial Papain and its Analysis, 444
Adler, L., Estimation of Amino-Acid and Polypeptide-Nitrogen in Barley, Malt, and Beer, by Titration with Acid after Treatment with Formaldehyde, 257
Agulhon, H. *See* Bertrand, G., *and* Agulhon, H.
Albright, A. R., Hydrogen Value of Some Essential Oils and Essential Oil Products. 1. Oils of Sassafras, Anise, Fennel, Clove, and Pimento, 557
Alder, A., *and* Coolbaugh, M. F., Volumetric Estimation of Lead, 373
Alleman, G., Quantity and Character of Creosote in Well-Preserved Timbers, 496
Allen, H. C., Electrolytic Reduction of Iron for Permanganate Titration, 328
Aloy, J., *and* Rabaut, C., Detection of Morphine and Phenols by Means of Uranium Salts, 438
Ambronn, H., Examination of Nitrocelluloses with the Polarising Microscope, 94
Ames, J. W., *and* Gaither, E. W., Estimation of Carbon in Soils and Soil Extracts, 449
Amos, S. L. *See* Day, F. E., *and* Amos, S. L.
Anderson, A. A., Furfural in Cider Vinegar, 256
———, V. G., Influence of Weather Conditions upon the Amounts of Nitric Acid and of Nitrous Acid in the Rainfall, 501
Andrews, E. R. *See* Coste, J. H., *and* Andrews, E. R.
Angerhausen, J. *See* Berg, P., *and* Angerhausen, J.
———. *See also* Nottbohm, F. E., *and* Angerhausen, J.
Appleby, M. P., Determination of Viscosity, 104
Arendt, O., Shaking Apparatus, 284
Armani, G., *and* Barboni, J., Analysis of Adhesive Soluble Substances, 252
Arnold, H., Analysis of High-Grade Metallic Tungsten, 505
———, Analysis of Pyrophoric Alloys of Cerium and Iron, 415
———, W., Butter Fat Content of Margarine, 216
———, Detection of Sesame Oil in Margarine Containing Colouring Substances, 86
Arny, H. V., *and* Schaefer, H. H., Estimation of Casein by Means of Ferric Ammonium Sulphate, 496
Asch, D. *See* Asch, W., *and* Asch, D.
Asch, W., *and* Asch, D., The Silicates in Chemistry and Commerce, Including the Exposition of a Hexite and Pentite Theory of a Stereo-Chemical Theory of General Application, *Review*, 201
Atkins, W. R. G., Guaiacum Resin as a Reagent for the Detection of Oxydases and of Minute Traces of Copper, 39
Atkinson, H. M. *See* Boyd, J. G., *and* Atkinson, H. M.
Auerbach, F., *and* Pick, H., Action of Sodium Hydrogen Carbonate Solutions on Difficultly Soluble Lead Salts, 143
———, Behaviour of Lead Carbonate, Basic Lead Carbonate, and Lead Sulphate towards Alkali Carbonate Solutions, 53

Azadian, A., Estimation of Hæmatin in Blood, 262
————, Estimation of the Alkaloids: Application to Preparations of Nux Vomica, 122

Bach, A., Delicacy of the Peroxydase Reaction, 402
Bachmann, F., New Seal for Prevention of Aeration in De-aerated Liquids, 509
Bacon, C. W. *See* Rosanoff, M. R., Bacon, C. W., *and* White, R. H.
———, R. F. *See* Brooks, B. T., *and* Bacon, R. F.
———, W. *See* Tindall, R. W., *and* Bacon, W.
Bailey, E. M., Some Reactions of Chrysophanic Acid, with Reference to its Detection in Complex Medicinal Preparations, 353
———, L. H., Non-Uniformity of Drying-Oven Temperatures, 457
Baker, J. L., Reports to the Local Government Board: II. On the Analysis and Composition of Some Proprietary Foods for Infants, 464
———, R. T., *and* Smith, H. G., Correlation between the Specific Characters of the Tasmanian and Australian Eucalypts, 435
Ball, T. R., *and* Smith, G. M., Volumetric Estimation of Titanium by Means of Ferric Chloride, 563
Bang, J., Micro-Estimation of Sugar in Blood, 90
Bannister, C. O., *and* Du Vergier, E. A., The Determination of Iridium in Platinum-Iridium Alloys, 340
Barger, G., The Simpler Natural Bases, *Review*, 574
Baragiola, W. I., *and* Godet, C., Method of Expressing Results of Wine Analyses according to the Latest Chemico-Physical Principles, 127
——————, *and* Schuppli, O., Estimation of Lactic Acid in Wine by Moslinger's Barium Chloride Method, 397
——————. *See* Von der Heide, C., *and* Baragiola, W. I.
Barbieri, N. A., Proximate Analysis of Wheat, 441
Barboni, J. *See* Armani, G., *and* Barboni, J.
Bardèt, J. *See* Boulanger, C., *and* Bardèt, J.
Barnebey, O. L., Permanganate Titration of Iron in Presence of Chlorides, 415
——————, Silver-Plated Copper Gauze Electrode in Zinc Determination, 382
Barry, F. *See* Richards, T. W., *and* Barry, F.
Barthel, C., *and* Sondén, K., Detection of Coconut Oil in Butter Fat by Means of Polenske's Distillation Method and Bömer's Phytosteryl Acetate Test, 254
Barton, A W. *See* Long, J. H., *and* Barton, A. W.
Baskerville, C., Apparatus for Taking Dust and Bacteria Samples of Air, 283
Baur, E., *and* Trümpler, G., Colorimetric Estimation of Creatine, 309
Baxter, G. P., Convenient Standard End-Point in Titration of Alkali Carbonates with Hydrochloric Acid, 268
—————, *and* Grover, F. L., Resistance of Platinum Vessels to Hot Nitric Acid, 382
Beadle, C., *and* Stevens, H. P., Apparatus for the Drying of Paper for Test Purposes, 508
——————————, Dry and Wet Strengths of Paper and Paper Yarns, 322
Beam, W., Determination of Humus in Heavy Clay Soils, 132
Beckett, E. G., Determination of Nitrogen in Gun-cotton by Means of the Nitrometer, 409
Bedford, M. H., *and* Pfanstiel, R., Method for the Estimation of Zinc in Treated Wood, 564
Béhal, A., Analysis of Essential Oils, 396
Belencki, E. *See* Woker, G., *and* Belencki, E.
Bender, A. W., Estimation of Mercuric Iodide in Tablets, 493
Bennett, C. W., Kenny, H. C., *and* Dugliss, R.P., Electrolytic Separation of Nickel, 454

Berg, P., *and* Angerhausen, J., Unsaponifiable Constituents of Mowrah Fat and the Detection of this Fat in Animal and Vegetable Fats, 310
———, Unsaponifiable Constituents of Shea Butter and their Relation to those of Mowrah Fat, 440
Bernhardt, —, Determination of Early Stages of Pregnancy, 177
Bertrand, G., *and* Agulhon, H., Estimation of Boric Acid in Foods, 123
———, Method of Estimating Very Small Quantities of Boric Acid in Organic Substances, 96
———, Occurrence of Borid Acid in Milk and in Eggs, 123
Besson, A. A., Estimation of Fatty Acids in Soaps, 311
Bhaduri, K., Essential Oil of *Ocymum Pilosum, Roxb.*, 438
Bianchi, A., *and* Malatesta, G., Waxes from Flax and Hemp, 560
Bianchini, G. *See* Canzoneri, F., *and* Bianchini, G.
Bingham, E. C., New Viscometer for General Scientific and Technical Purposes, 284
Bizzel, J. A., *and* Lyon, T. L., Estimation of the Lime Requirement of Soils, 90
Blair, H. *See* Collins, S. H., *and* Blair, H.
Bland, N., *and* Lloyd, L. L., Estimation of Aldoses (Part I.), Action of Neutral Sodium Hypoiodite, 556
Blanksma, J. J., Detection of Methyl Alcohol and Ethyl Alcohol, 184
Bloor, W. R., Nephelometric Method for the Determination of Fat in Milk, 356
Blount, B. *See* Woodcock, W. H., *and* Blount, B.
Böcker, E., Determination of the Hydrocarbon Content of Concentrated Lemon Oils, 185
Bode, G., *and* Wlokka, A., New German Standards for Malt Analysis, 436
Bodnár, J., Detection and Approximate Estimation of Traces of Thiosulphates in Sulphites, 147
———, Estimation of Thiosulphate in Presence of Sulphite, 101
Bömer, A., Krönig, R., Ley, R., *and* Merten, H., Detection of Beef-Fat in Lard, 171
———, Limprich, R., Krönig, R., *and* Kuhlmann, J., Detection of Beef-Fat in Lard, 84
Bonis, —, Composition of Rums, 401
Bornemann, E., Extraction of Finely Powdered Substances, 420
Bosshard, E., *and* Huggenberg, W., Estimation of Free Alkali in Soaps, 140
Boswell, M. C., Estimation of Oxygen in Organic Compounds, 135
Bosworth, A. W., Use of Sodium Citrate for Determination of Reverted Phosphoric Acid, 276
Boulanger, C., *and* Bardèt, J., Presence of Gallium in Commercial Aluminium, 53
Bourion, F., *and* Sénéchal, A., Estimation of Chromium by Oxidation in Alkaline Solution, 143
Boyd, J. G., *and* Atkinson, H. M., Simple Thermo-Regulator, 63
Brady, W., Determination of Carbon in Steel by the Direct Combustion Method, 560
Brandt, L., Application of Diphenylcarbohydrazide (Diphenylcarbazide) as Indicator in Titration of Iron with Dichromate, 91
———, Destruction of Organic Matter in Bog Iron Ore, 372
———, Quantitative Separation of Arsenic from the Metals by Means of Hypophosphorous Acid, 269
Bridgman, J. A. *See* Lundell, G. E. F., *and* Bridgman, J. A.
Brock, F. P. *See* Redman, L. V., Weith, A. J., *and* Brock, F. P.
Brooks, B. T., *and* Bacon, R. F., Fluorescence of Petroleum Distillates, 446
Brown, A. L., Quantitative Determination of Body and Solvent in Varnish, 560
Browning, K. C., *and* Symons, C. T., Alcohol Estimation in the Tropics, 480
Brubaker, H. W., Some Natural Indicators, 499
Brunck, O., Estimation of Nickel by Means of Dimethylglyoxime, 375

Brunt, C. Van, Volumetric Estimation of Titanium and Chromium by Means of a Modified Reductor, 419

————, Nitrogen Generator, 422

Buchner, G., *and* Deckert, R., Commercial Analysis of Beeswax, 123

Budde, C., Extraction Apparatus Specially Designed for Solvents of High Specific Gravity, 240

Bühn, T. *See* Kirpal, A., *and* Bühn, T.

Bunzel, H. H., *and* Haselbring, H., Thermo-Regulator for Electrically Regulated Constant Temperature Chambers, 336

Burberg, W. *See* König, J., *and* Burberg, W.

Burgess, G. K., *and* Sale, P. D., Thermo-Electric Method for the Determination of the Purity of Platinum Wire, 381

Burgwin, J. H. K., Relation of Composition of Ash in Coal to its Fusing Temperature, 445

Burian, J., *and* Juránek, J., Rational Analysis of Clays, 499

Burke, C. E., *and* Scalione, C. C., Investigations on Oil of Black Sage, 559

Burn, J. H., Herzig and Meyer's Reaction Applied to Proteins and Amino-Acids, 317

Burnebey, O. L., Detection of Cyanides in Presence of Ferro and Ferricyanides and Thiocyanates, 365

Burrell, G. A., *and* Seibert, F. M., Errors in Gas Analysis Due to Assuming that the Molecular Volumes of All Gases are Alike, 231

————, Gas Analysis by Fractional Distillation at Low Temperatures, 414

Burrows, G. J., Inversion of Cane Sugar by Acids in Water-Alcohol Solutions, 323

Busch, M., Estimation of Halogens in Organic Compounds, 367

Busvold, N., Gravimetric Estimation of Nitrites, 145

Cain, J. R., Determination of Carbon in Steel and Iron by the Barium Carbonate Titration Method, 373

Cammidge, P. J., The Fæces of Children and Adults, *Review*, 242

Campbell, E. D. *See* Carney, R. J., *and* Campbell, E. D.

————, F. H., New Method for the Determination of Vapour Pressures and an Examination of a Source of Error in Certain Dynamical Methods, 511

Campos, N., Detection of Saponin in Aerated Waters, 440

Canzoneri, F., *and* Bianchini, G., Rancidity of Olive Oil and Oxidation of Oleic Acid in Presence of Sunlight, 255

Carles, P., Occurrence of Fluorine in Reagents, 231

Carletti, O., New Indicator for Use in Determining End-Point in Titrations with Fehling's Solution, 133

Carlson, J., Solubility of Atmospheric Oxygen in Water, 55

Carney, R. J., *and* Campbell, E. D., Estimation of Thorium in Monazite Sand, 378

Carré, P. *See* Hanriot, M., Carré, P., Seyewetz, A., Charabot, E., *and* Hébert, A.

Carter, F. G., Method by which the Corrected Sikes's Tables in Use in India, for Determining the Proof Strengths of Spirits at Temperatures from 40° F. to 100° F. by Sikes's Hydrometer, were Calculated, 532

Cavaignac, H., Precipitation of Aluminium in Presence of Fluorides, 269

Ceccherelli, A. *See* Tortelli, M., *and* Ceccherelli, A.

Chandelon, T., Analysis of Waste Acids from Nitrocellulose Manufacture, 183

Chapin, R. M., Preparation of Dry Starch, Soluble in Cold Water, for Use as an Indicator, 451

Chapman, A. C., Nitrogenous Constituents of Hops, 492

Charabot, E. *See* Hanriot, M., Carré, P., Seyewetz, A., Charabot, E., *and* Hébert, A.

Charitschkow, K., Colour Reactions of Organic Compounds with Trichloroacetic Acid, 410

Chattopadhyay, P. C., Estimation of Volatile Oils in Aromatic Plants, Drugs, and Spices by the Tailameter, 35
Chelle, L., Estimation of Carbohydrates and Lactic Acid in Blood and Other Physiological Liquids, 551
Chlopin, W. *See* Tschugaeff, L., *and* Chlopin, W.
Chodat, R., *and* Kummer, R. H., Detection of Peptides in Urine by the *p*-Cresol Tyrosinase Reaction, 555
Christian, M., Disinfection and Disinfectants, *Review*, 66
Clark, W. M., Improved Thermo-Regulator, 104
Clausmann, P. *See* Gautier, A., *and* Clausmann, P.
Clotworthy, H. G. S. *See* Thompson, W. H., Wallace, J. A., *and* Clotworthy, H. G. S.
Cocking, T. T., *and* Kettle, J. D., Analytical Characters of Benzoin, 393
Cohen, J. B., Organic Chemistry for Advanced Students. Vol. II., *Review*, 106
Cole, H. I. *See* Gooch, F. A., *and* Cole, H. I.
——, S. W., Estimation of Lactose by the Copper-Iodide Method, 324
Collins, S. H., *and* Blair, H., The Rate of Liberation of Hydrocyanic Acid from Linseed, 70
Compagno, I., New Method for Electro-Analysis of White Metals, 52
Condelli, S., Detection and Estimation of Saccharin, 401
Coninck, W. O., de, Colour Reaction of Succinic and Malic Acids, 186
Cook, F. C., Distribution of Nitrogen in Vegetable, Yeast, and Meat Extracts, 444
Coolbaugh, M. F. *See* Alder, A., *and* Coolbaugh, M. F.
Corfield, C. E., *and* Pratt, W. R., Determination of Iron in Presence of Phosphoric Acid, 416
Coste, J. H., *and* Andrews, E. R., Fuel, Gaseous, Solid, and Liquid, *Review*, 575
Coutts, F. J. H., Reports to the Local Government Board: I. On the Use of Proprietary Foods for Infant Feeding, 464
Cramer, B. J. *See* Sinnatt, F S., *and* Cramer, B. J.
Cripps, R. A., *and* Wright, A. G., Note on the Presence of Sulphates in Flour, 429
Cumming, A. C., *and* Kay, S. A., A Textbook of Quantitative Chemical Analysis, *Review*, 240
Curtman, L. J., *and* Marcus, J. K., Separation of the Copper and Tin Groups, 371

Daish, A. J., Methods of Estimation of Carbohydrates: III. The Cupric-Reducing Power of the Pentoses—Xylose and Arabinose, 555
——. *See* Davis, W. A., *and* Daish, A. J.
Danckwortt, P. W., Estimation of Silver in Collargol and Organic Liquids, 278
Daniels, F. C. T., Estimation of Chromium and Manganese in Iron and Steel, 454
Darabsett, D. B. *See* Warth, F. J., *and* Darabsett, D. B.
Datta, R. L., Estimation of Semicarbazide by its Interaction with Halogens and Halogen Oxyacids, 323
Davis, W. A., *and* Daish, A. J., Estimation of Starch in Plant Material, 312
Day, A. A. *See* Kendall, A. I., Day, A. A., *and* Walker, A. W.
——, F. E., *and* Amos, S. L., Temperature Corrections to be Applied to the Specific Gravities of Malt Worts and Beers, 256
Dean, A. L. *See* Downs, C. R., *and* Dean, A. L.
Debourdeaux, —, Estimation of Morphine in Acid Opium Solutions, 31
Deckert, R. *See* Buchner, G., *and* Deckert, R.
Dede, L., Formation of Sediment in Bottled Mineral Waters. Estimation of Silica, Iron, and Aluminium in Mineral Waters, 126
Degrazia, J. von, Tobacco Resins, 220
Dehn, W. M., *and* Hartmann, F. A., Comparison of Various Preservatives for Urine, 177
————, Picrate Colorimetric Method for the Estimation of Carbohydrates, 180

Dehn, W. M., *and* Hartmann, F. A., "Urinod," the Cause of the Characteristic Odour of Urine, 554
———, Volatile Substances of Urine, 554
Deiss, E., Standardisation of Volumetric Arsenite Solutions, 271
Derby, I. H., *and* Marden, J. W., Method for Electrolytic Heating and Regulation of Thermostats, 64
Desgrez, A., *and* Moore, R., Estimation of Urea, 412
Dewey, F. P., Determination of Silver and Base Metal in Precious Metal Bullion, 502
Diedrichs, A. *See* Sprinkmeyer, H., *and* Diedrichs, A.
Dienert, F., New Nephelometer and its Use in Analysis, 283
Dieterich, K., Peru Balsam, 400
Dillon, T. *See* Ryan, H., *and* Dillon, T.
Doherty, W. M., Determination of Vanillin, 220
———, Estimation of Carbon Dioxide in Air, 324
———, Flame Test for Chloral Hydrate, 217
Donath, H. C. *See* Weiser, St., *and* Donath, H. C.
Dons, R., Reductase (Fermentation-Reductase) Test, 224
Dorner, A., Titration of Small Quantities of Carbonic Acid, 143
Doroschevski, A., *and* Dvorzanczyk, S., Applicability of Conductivity Methods in Examination of Natural Waters, 149
Dorta, G. *See* Fachini, S., *and* Dorta, G.
Dott, D. B., Estimation of Strychnine in Presence of Brucine, 394
Downs, C. R., *and* Dean, A. L., Composition of Water-Gas Tar, 369
Dowzard, E., Estimation of Camphor in Tablets and Pills, 353
Dreaper, W. P., Determination of Malt Extract, 407
Drummond, J. C. *See* Rosenheim, O., *and* Drummond, J. C.
Dubois, W. L., Report on Cocoa and Cocoa Products, 123
Dubovitz, H., Estimation of the Weight of a Substance by Titration, 194
Dubrisay, R., New Physico-Chemical Volumetric Method, 330
Dubsky, J. V., Quantitative Micro-Elementary Analysis of Organic Substances by F. Pregl's Methods, 280
Duccini, C., New Method of Estimating Mercury, 144
Dufty, L., Estimation of Cobalt in High-Speed Steels, 503
Dugliss, R. P. *See* Bennett, C. W., Kenny, H. C., *and* Dugliss, R. P.
Dupré, H. A. *See* Lynde, C. J., *and* Dupré, H. A.
Durand, H., Estimation of Saccharin in Foods, 86
Du Verdier, E. A. *See* Bannister, C. O., *and* Du Verdier, E. A.
Duyser, C. A., *and* Lewis, W. K., Method for Determining the Value of Disinfectants, 261
Dvorzanczyk, S. *See* Doroschevski, A., *and* Dvorzanczyk, S.

Ebler, E., Estimation of Radium by the Emanation Method, 58
Eck, J. J. van, Estimation of Dissolved Oxygen in Water, 55
Eckert, A., Estimation of Nitrogen in Nitro-Compounds by Kjeldahl's Method, 93
Edeleanu, L., Separation of Hydrocarbons by Means of Liquid Sulphur Dioxide, 321
Edwards, J. D., Estimation of Ammonia in Illuminating Gas, 369
Eegriwe, E., Sensitiveness of Various Reagents for Lead, 374
Egerton, A. C., Heat Test of Explosives, 184
Eijkman, C., Fermentation Test at 46° C. as an Aid in Water Examination, 129
Ellms, J. W., *and* Hauser, S. J., Effect of Ferric Salts and Nitrites on the *o*-Tolidine and Starch Iodide Tests for Free Chlorine, 454
———, Orthotolodine as Reagent for Colorimetric Estimation of Small Quantities of Free Chlorine, 46
Elsdon, G. D., The Composition of Palm-Kernel Oil, 78

Elsdon, G. D. *See* Liverseege, J. F., *and* Elsdon, G. D.
Emery, W. O., Estimation of Acetanilide and Phenacetin in Admixture, 433
———, *and* Palkin, S., Estimation of Antipyrin, 480
Engeland, R., Detection and Estimation of Monamino-Acids, 316
Engler, C., Sieveking, H., *and* Koenig, A., Fontactoscope for Measuring the Radio-Activity of Spring Waters, 423
———, Measurement of Radio-Activity of Springs, 277
Erban, F., Estimation of Riçinoleic Acid in Oils, 410
Eskew, M. H. *See* Miller, E. R., Taylor, G. W., *and* Eskew, M. H.

Fachini, S., *and* Dorta, G., Note on the Fatty Acids and Detection of Arachidic Acid, 122
Fahrion, W., Analysis of Turkey-Red Oil, 48.
Fairlie, A. M., Pitot-Tubes for Measurement of Gas Velocities, 456
Falk, K. G., *and* Sugiura, K., Experiments with Castor Bean Urease (Studies on Enzyme Action, XI.), 553
Fanto, R., Detection of Small Quantities of Rye Flour in Wheat Flour, 400
Farbell, R. F., Estimation of Arsenic in Hydrochloric and Sulphuric Acids, 370
Feder, E., Polarimetric Estimation of Lactose in Milk, 399
Fegely, W. H. *See* Lee, R. E., Trickey, J. P., *and* Fegely, W. H.
Feild, A. L., Electrical Contact Vapour-Pressure Thermoregulator, 154
Fenaroli, P., Essential Oil of *Citrus Sinensis*, 558
Fendler, G., *and* Stüber, W., Estimation of Caffeine in Coffee, 394
Fincke, H., Detection of Formaldehyde and of Formaldehyde Compounds by Means of Schiff's Reagent containing Hydrochloric Acid, 172
Finzi, C., Organic Phosphoric Acid Compounds in Concentrated Musts and Wines, 398
Fischer, K., *and* Wewerinke, J., Detection of Beef Fat in Lard by Polenske's and Bömer's Methods, 216
Fleissig, —, Bernadot's Vinometer, 457
Fletcher, A. L., Estimation of Radium in Radio-Active Substances, 57
Florentin, D. See Kling, A., Genin, V., *and* Florentin, D.
Foehr, C. *See* Wüstenfeld, H., *and* Foehr, C.
Forbester, R. E. *See* Paterson, J. H., *and* Forbester, R. E.
Fornet, A., Rapid Estimation of Water in Foods and Feeding Stuffs, 36
Fosse, R., Gravimetric Estimation of Urea in Urine, 362
Identification of Urea and its Precipitation from very Dilute Solutions, 48
----, Quantitative Gravimetric Estimation of Urea, 268
Fox, P. J., Titration of Calcium and Magnesium in the Same Solution, 51
François, M., Estimation of Caffeine in Kola Preparations, 31
———, Extraction Apparatus, 61
Frankforter, G. B., New Apparatus for Estimation of Hydrogen Sulphide in Water. Part I., 457
Freese, —. *See* Sisley, P., *and* Freese, —.
Fresenius, R., *and* Grünhut, L., Apparatus for the Approximate Estimation of Free Carbon Dioxide in Mineral Waters, 238
Freund, M., Simple Extraction Apparatus, 381
Frey, O., Valuation of Pepsin, 130
Friedmann, A., Analyses of Waters from Thermal Springs in Palestine, 102
Friedrichs, F., Modified Lothar Meyer's Apparatus for the Purification of Mercury, 151
Fritzsche, M., Detection of Phytosterol in Animal Fats, 84
Frost, W. D., Pipettometer, 509
Furlong, J. R., The Estimation of Hydrocyanic Acid in Feeding-Stuffs, and its Occurrence in Millet and Guinea Corn, 430

Gaither, E. W. *See* Ames, J. M., *and* Gaither, E. W.
Gardner, H. A., Composition of Paint Vapours, 265
Garn, W. von. *See* Meyer, J., *and* Garn, W. von.
Gaunt, R., Viscosity of Rubber Solutions, 322
Gautier, A., and Clausmann, P., Fluorine in Mineral Waters, 401
Gauvin, R., *and* Skarzynski, V., Rapid Estimation of Different Sulphur Compounds in Urine, 101
Geake, J. J. *See* Lander, G. D., *and* Geake, J. J.
Gelin, E. *See* Kling, A., *and* Gelin, E.
Genin, V. *See* Kling, A., Genin, V., *and* Florentin, D.
Gemmell, A., The Insoluble Bromide Value of Oils and its Determination, 297
Gerasimov, A., High Temperature Thermostat, 153
Germann, F. E. E. *See* Guye, P. A., *and* Germann, F. E. E.
Gerum, J., Estimation of Ivory-Nut Meal in Flours and Fodders, 218
Gilbert, H. N., Manostat for Use in Gas Analysis, 458
Gillet, R., Comparative Study of Different Methods of Determining Cane-Sugar in Beet Products by the Double Polarisation Process, 435
Gillett, H. W., *and* Norton, A. B., Approximate Melting-Points of Some Commercial Copper Alloys, 98
Glauser, R., Tellurium Dioxide for Combustion Analyses, 192
Gluud, W., *and* Kempf, B., Stirring Apparatus, 422
Godet, C. *See* Baragiola, W. T., *and* Godet, G.
Gooch, F. A., *and* Cole, H. I., Use of Telluric Acid in Estimation of Bromine Associated with Chlorine in Haloid Salts, 331
————, Reckert, F. C., *and* Kuzirian, S. B., Dehydration and Separation of Silica, 190
Goodnow, E. H. *See* Tolman, L. M., *and* Goodnow, E. H.
Gori, G., Use of Carbon Tetrachloride in the Extraction and Estimation of the Active Principles of Drugs and Medicinal Plants, 255
Gortner, R. A., Device to Assist Filtration by Suction, 508
Gothe, F., Colorimetric Estimation of Iron in Water, 333
Goujon, —. *See* Marchadier, —, *and* Goujon, —.
Gowing-Scopes, L., The Properties of Some Chlorhydrocarbons and their Uses in Chemical Analysis, 4, 385
Goy, S., Gravimetric Estimation of Calcium as Oxalate, 50
Graber, H. T., Assay of Digestive Ferments, 359
————, Malt Extract, 356
Graham, J. I., *and* Winmill, T. F., Estimation of Carbon Monoxide, 445
Graves, S. A., *and* Kober, P. A., "Tricresol" as a Substitute for Toluene in Enzyme Work, 263
————. *See* Kober, P. A., *and* Graves, S. A.
Gray, F. W., Adiabatic Colorimeter, 281
Gray, T., Automatic Gas-Sampling Apparatus, with Some Observations on Sampling, 103
Grey, E. C., Volumetric Estimation of Carbon in Aliphatic Substances in the Wet Way, 556
Griesbach, W., *and* Straszner, H., Estimation of Sugar in Blood, 90
Griese, E., Detection of Minute Quantities of Phlorrhizin Alone and in Presence of Phlorin, 410
Griffin, M. L., Chemical Valuation of Wood for Pulp, 451
Grimbert, L., *and* Laudat, M., Estimation of Lipoids in Blood-Serum, 176
Grimme, C., A Skilfully Adulterated Cacao Butter, 216
————, Cacao Butter Substitute, 434
————, Estimation of Husks in Cottonseed Cakes and Meals, 173

Grimwood, R. G., Analysis of Crude Glycerol by the International Standard Methods, 1911. Estimation of Organic Residue, 34
Grossfeld, J., Estimation of Crude Fibre in Finely Powdered Substances, 218
————. *See* König, J., *and* Grossfeld, J.
Grover, F. L. *See* Baxter, G. P., *and* Grover, F. L.
Grube, G., *and* Krüger, J., Volumetric Estimation of Cyanamide, 365
Grünhut, L. *See* Fresenius, R., *and* Grünhut, L.
Grützner, R., Estimation of Iodine in Organic Substances, 367
Guareschi, I., Detection of Bromides in Presence of Thiocyanates and Ferrocyanides, 187
————, Detection of Bromides in Presence of Thiocyanates and Ferrocyanides: Action of Chromic Acid on Cyanogen Bromide, 412
————, Detection of Bromine in Gases and Vapours. VII. Communication, 271
Gude, K. *See* Pinoff, F., *and* Gude, K.
Guérin, G., Identification of Traces of Strychnine by Means of Manganese Carbonate, 357
Gutbier, A., *and* Huber, J., Gravimetric Estimation of Tellurium, 379
————, *and* Weise, G. L., Separation and Estimation of Tungstic Acid, 379
Gutmann, S., *and* Schlesinger, F., Estimation of Chlorine in Blood Serum, 223
Guye, P. A., *and* Germann, F. E. E., Analysis of Very Small Quantities of Gas: Application to Air Analysis, 413

Haar, A. W. van der, Estimation of Benzoyl in Benzoyl Derivatives, 405
Haber, F., Fire-Damp Whistle, 193
Hackl, O., Separation of Iron and Manganese, 272
Haglund, E. *See* Weigmann, —, *and* Haglund, E.
Hahn, A., Vacuum-Jacketed Thermometer, 154
Hall, C., *and* Howell, S. P., Tests of Permissible Explosives (Report), 286
Halla, F., Separation of Calcium from Magnesium, 142
Hanriot, M., Carré, P., Seyewetz, A., Charabot, E., *and* Hébert, A., Principes d'Analyse et de Synthèse en Chimie Organique, *Review*, 383
Hansson, N., Method for Calculating the Value of Foods from the Point of View of Milk Production, 171
Harding, H. G. A., Notes on the Determination of the Cineol Content of Eucalyptus Oils, 475
Harries, C., Physical Constants of Isoprene, 406
Hart, E. B., *and* Lamb, A. R., Occurrence of Methyl Alcohol in Corn Silage, 549
Hart, F., Estimation of Blast-Furnace Slag in Portland Cement, 187
Hartmann, F. A. *See* Dehn, W. U., *and* Hartmann, F. A.
Hartung, E. J., New Method for Determining the Specific Heats of Liquids, 510
Hartwig, L., *and* Schellbach, H., Colorimetric Estimation of Small Quantities of Manganese in Water, 54
Haselbring, H. *See* Bunzel, H. H., *and* Haselbring, H.
Hassler, C. *See* Heuser, G., *and* Hassler, C.
Hauser, E., Detection and Estimation of Gaseous Hydrocarbons in the Water of Mineral Springs, 227
———, S. J. *See* Ellms, J. W., *and* Hauser, S. J.
Haussding, F., Estimation of Citric Acid Soluble Phosphoric Acid in Basic Slag, 377
Häussler, E. P., Colour Reactions for Citric Acid, 395
————, Composition of the Radicles of Cacao Beans, 308
————, Reactions of Vanillin, 315
Hayden, E. M., jun. *See* Thornton, W. M., jun., *and* Hayden, E. M., jun.
Hazen, A., Clean Water and How to Get It, *Review*, 473
Hébert, A. *See* Hanriot, M., Carré, P., Seyewetz, A., Charabot, E., *and* Hébert, A.

Heide, C. von der, *and* Baragiola, W. I., Difference between the Proportion of Acid and the Degree of Acidity of Wine, 221
Heidenhain, M., Treatment of Sinews, particularly by the Application of Ruthenium Red and the Mallory Connective Tissue Stain, 89
Heiduschka, A., Bromine Thermal Value of Turpentine Oil, 314
Heinze, R., Apparatus for Estimation of Small Quantities by Electrolysis with the Rotating Cathode, 334
Henry, J. A., The Plant Alkaloids, *Review*, 104
Herzberg, W., Identification of Rosin and Glue Sizings, 411
Herzfeld, E., Estimation of Tryptophane, 47
Heubner, W., Estimation of Phosphoric Acid in the Presence of Phosphoric Acid Esters, 559
———, *and* Stadler, H., Volumetric Method for the Estimation of Phytin, 560
Heuser, G., *and* Hassler, C., Detection of Extracted Paprika, 174
Hevesy, G. von. *See* Paneth, F., *and* Hevesy, G. von
Hibbard, P. L., Study of Pemberton-Kilgore Method for Determination of Phosphoric Acid, 100
Hill, C. A., Pharmaceutical Chemical Standards, 439
Hintz, E., Estimation of Iron in Iron Ores, 144
Holde, D., *and* Meyerheim, G., Estimation of Asphaltic Matters in Dark Mineral Oils, 178
Holden, H. C., *and* James, C., Separation of Yttrium from the Yttrium Earths. Part I., 280; Part II., 420
Holland, E. B., Estimation of the Acetyl Value of Oils, Fats, etc., 362
Holmberg, B., Titration of Potassium Xanthate, 95
Holmes, A. *See* Jacobson, C. A., *and* Holmes, A.
Hondovsky, H., Estimation of Allantoin in Urine, 263
Hottenroth, V., Method of Estimating Formic Acid, 320
Hottinger, R., "Lacmosol," the Active Constituent of Lacmoid, 556
———, Modifications of the Kjeldahl Method of Estimating Nitrogen, 227
Hough, A. T., Application of Tungsten Salts to the Analysis of Tanning Materials, 507
Houston, A. C., Metropolitan Water Board. Tenth Research Report, 514
Howell, S. P. *See* Hall, C., *and* Howell, S. P.
Huber, J. *See* Gutbier, A., *and* Huber, J.
Hudson, C. S., Method of Preparing Strong Invertase Solutions from Top and Bottom Yeasts, 443
Huggenberg, W. *See* Bosshard, E., *and* Huggenberg, W.
Hunder, M., *and* Suleimann, A., Solubility of Silica and Rendering Silica Insoluble in Acid Solutions, 191
Hutchinson, H. B., *and* MacLennan, K., Determination of Lime Requirements of Soil, 450
———, Determination of Soil Carbonates, 562
Hüttner, C., Colorimetric Estimation of Cobalt, Nickel, Iron, and Copper, 326

Ipiens, A., Note on Lux's Method for the Estimation of Lead Peroxide, 328
Isnard, E., Detection of Picric Acid in Urine, 224
Itano, A. *See* Suchtelen, F. H. H. van, *and* Itano, A.
Ivanov, V. N., New and Sensitive Reaction of Lead, 274

Jackson, L. C., McNab, L., *and* Rothera, A. C. H., Electrical Conductivity of Milk. Electrometric Determination of End-Point in Manufacture of Sweetened Condensed Milk, 174
Jacobsohn, F., Estimation of Loading in Caoutchouc Mixtures according to the Standards of the Verband deutscher Elektrotechniker, 91

Jacobson, C. A., *and* Holmes, A., Enzymes Present in Alfalfa (Lucerne), 552
James, C. *See* Holden, H. C., *and* James, C.
———. *See also* Morgan, J. C., *and* James, C.
———. *See also* Smith T. O., *and* James, C.
Jamieson, G. S., *and* Wrenshall, R., Estimation of Titanium as Phosphate, 279
Jaubert, G. F., Analysis of "Silicol" and Other Silicon Compounds Used for the Production of Hydrogen, 147
Jenkins, W. M., Estimation of Podophyllum Resin, 439
Joassart, N. *See* Koninck, L. L. de, *and* Joassart, N.
Johnstone, S. J., Analysis of Monazites, 189
Jolles, A., Estimation of Urea, 95
Joly, J., Temperature of Sublimation, 153
Joseph, A. E., *and* Rae, W. N., Notes on Alcoholometry, II., 532
Juránek, J. *See* Burian, J., *and* Juránek, J.

Kantorowicz, H., Analysis of Petroleum Oil and Paraffin Wax, 42, 266
———, Apparatus for Testing the Volatility or Oxidisability of Oils, 334
Kay, S. A. *See* Cumming, A. C., *and* Kay, S. A.
Kedesdy, E., Volumetric Estimation of Sulphurous Acid, 331
Kempf, R. *See* Gluud, W., *and* Kempf, R.
Kendall, A. I., Day, A. A., *and* Walker, A. W., Studies in Bacterial Metabolism in Milk, 494
Kenny, H. C. *See* Bennett, C. W., Kenny, H. C., *and* Dugliss, R. P.
Kerr, R. H., Detection of Nickel in Fats, 254
Kettle, J. D. *See* Cocking, T. T., *and* Kettle, J. D.
Kirpal, A., *and* Bühn, T., Estimation of Methoxyl in Compounds containing Sulphur, 265
Klein, R., *and* Reinau, E., Carbon Dioxide and Plants, 260
Klimont, J., *and* Meisl, E., Characteristics of Stag's Fat, 32
Kling, A., *and* Gelin, E. Separation and Estimation of Beryllium, 232
———, *and* Lassieur, A., Physico-Chemical Estimation of Sulphates, 191
———, Genin V., *and* Florentin, D., Estimation of Free Alkali in Commercial Soda and Soaps by the Barium Chloride Method, 229
Klinger, M.,Determination of the Softening Point of Pitch, 136, 321
Klostermann, M., Detection of Vegetable Fats in Animal Fats, 32
———, *and* Opitz, H., Estimation of Cholesterol in Fats, 310
Knapp, A. W., Gru-Gru Oil, 124
Knecht, E.. Volumetric Estimation of Titanium, 102
Kober, P. A., *and* Graves, S. A., Nephelometry in the Study of Nucleases, 359
———. *See* Graves, S. A., *and* Kober, P. A.
Koch, G. P. *See* Thatcher, R. W., *and* Koch, G. P.
Koehler, A., Marqueyrol, M., *and* Loriette, H., Estimation of Nitrogen in Nitrates and Nitrocelluloses, 276
Koenig, A. *See* Engler, C., Sieveking, H., *and* Koenig, A.
Koepping, E. D., Analysis of Copper, Tin, and Silicon Alloys, 500
König, J., *and* Burberg, W., Separation and Estimation of Sugars in Gingerbread and Similar Products, 358
———, *and* Grossfeld, J., Composition of the Soft Roe and Hard Roe of Fishes, 261
Koninck, L. L. de, *and* Joassart, N., Estimation of Ferrocyanide by Titration with Alkali Bromate, 366
Kooper, W. D., Determination of the Viscosity of Milk as a Means of Detecting the Addition of Water, 400
Krais, P., Industrial Applications of Various Processes and Systems for the Measurement and Nomenclature of Colour Shades, 149

Kratzmann, E., Micro-Chemical Detection of Aluminium, 269
Kreidl, A., *and* Lenk, E., Fat Content of Milk and Rate of Coagulation with Rennet, 357
Kreusler, U., Gas Generator for Permanent Use, 239
Krönig, R. *See* Bömer, A., Krönig, R., Ley, R., *and* Merten, H.
———— *See* Bömer, A., Limprich, R., Krönig, R., *and* Kuhlmann, J.
Kropat, K., Rapid Estimation of Fat in Cheese, Butter, and Cream, 253
Krüger, J. *See* Grube, G., *and* Krüger, J.
Krumbhaar, —, Gravimetric Estimation of the Oxygen Absorption of Linseed Oil during Drying, 92
Krzizan, R., Analysis of Saffron, 312
Kuhlmann, J. *See* Bömer, A., Limprich, R., Krönig, R., *and* Kuhlmann, J.
Kulisch, P., Malt Wine, 128
Kummer, R. H. *See* Chodat, R., *and* Kummer, R. H.
Kunzel, E. *See* Schönfeld, F., *and* Kunzel, E.
Kuzirian, S. B., Application of Sodium Paratungstate and the Blowpipe Flame to the Estimation of the Acid Radicals in Chlorides, Chlorates, Perchlorates, Bromides, Bromates, and Fluorides, 188
————, Correcting Silica for Occluded Salts, 191
————, Estimation of Water of Crystallisation in Sulphates, 193
————. *See* Gooch, F. A., Reckert, F. C., *and* Kuzirian, S. B.

Lamb, A. B., *and* Lee, R. E., Densities of Certain Dilute Aqueous Solutions by a New and Precise Method, 61
——, A. R. *See* Hart, E. B., *and* Lamb, A. R.
——, M. C., Analysis of the Saline Matter Adhering to the Flesh Side of Wet Salted East India Goat Skins, 402
Lander, G. D., *and* Geake, J. J., Iodimetry of Arsenic, Copper, and Iron, 116
————————, The Detection of Castor Seeds, 292
Lassieur, A. *See* Kling, A., *and* Lassieur, A.
Laudat, M. *See* Grimbert, L., *and* Laudat, M.
Lavers, H., Effect of Tungsten on Ammonium Molybdate Assay for Lead, 192
Leather, J. W., The Detection of Added Water in Milk, 432
Lederle, P. *See* Mach, F., *and* Lederle, P.
Lee, R. E., Trickey, J. P., *and* Fegely, W. H., Method for Rapid Quantitative Analysis of Bronze and Brass, 453
————. *See* Lamb, A. B., *and* Lee, R. E.
Lehmann, F., Estimation of Silver in Colloidal Silver, 279
Leiste, —, *and* Stiepel, C., Method of Estimating Colophony in Fats and Soaps, 181
Leitch, H. W., Test for Silicates in Soap, 550
Leitmeier, H., Determination of Melting-Points of Silicates, 236
Lematte, L., Estimation of Amino-Acids in Blood, 315
Lenk, E. *See* Kreidl, A., *and* Lenk, E.
Letts, E. A., *and* Rea, F. W., Colorimetric Method for Detecting and Estimating Nitrates and Nitrites, 276
————————, On Fresenius's Method for Determining Small Quantities of Nitrites, and its Sensitiveness compared with the *m*-Phenylene-Diamine Reaction, 350
Levallois, F. *See* de Vilmorin, P., *and* Levallois, F.
Levene, P. A., *and* Slyke, D. D. van, Separation of *d*-Alanine and *d*-Valine, 178
Levy, L., Estimation of Total Sulphurous Acid in Wine, 87
Lewis, E. A., Rotating Cathode and Anode for Rapid Electrolytic Analysis, 334
——, W. K. *See* Duyser, C. A., *and* Lewis, W. K.
Ley, R. *See* Bömer, A., Krönig, R., Ley, R., *and* Merten, H.

Limprich, R. *See* Bömer, A., Limprich, R., Krönig, R., *and* Kuhlmann, J.
Lindet, L., Estimation of "Cellulose" in Flour as a Measure of the Degree of Bolting, 309
Lindt, V., Colorimetric Estimation of Nickel, 327
Ling, A. R., Detection of Raw Beet Products in Admixture with Raw Cane Products, 258
Liverseege, J. F., *and* Elsdon, G. D., Notes on "Sharps," 251
Lloyd, L. L. *See* Bland, N., *and* Lloyd, L. L.
Löb, W., *and* Prorsk, A., Manometric Method for the Estimation of Urea, 553
Loebell, H., Analysis of Asphalts, 131
Loevy, J., Loss of Noble Metals on Cupellation, 145
Lohnis, F., Titration of Milk with Alcohol at Different Degrees of Concentration, 317
Long, J. H., *and* Barton, A. W., Comparison of Methods for the Determination of the Proteolytic Activity of Pancreas Preparations, 551
Loriette, H. *See* Koehler, A., Marqueyrol, M., *and* Loriette, H.
Losanitsch, M., Electrolytic Estimation of Mercury in Fulminate, 275
Löwe, F., Sugar Refractometer, 59
Lucion, R., Zeiss Interferometer for the Analysis of Gases, 103
Lüers, H., Electrical Conductivity of Beers, 352
Lührig, H., Colorimetric Estimation of Traces of Manganese in Water, 380
Lundell, G. E. F., *and* Bridgman, J. A., New Method for Determination of Hydrocyanic Acid and Alkali Cyanides, 448
Lunge, G., The Manufacture of Sulphuric Acid and Alkali, with the Collateral Branches, *Review*, 200
Lynde, C. J., *and* Dupré, H. A., Measuring the Capillary Lift of Soils, 318
Lyon, T. L. *See* Bizzel, J. A., *and* Lyon, T. L.

Mach, F., *and* Lederle, P., Commercial Copper Sulphate, 414
MacLean, J. S., Estimation of Pyruvic Acid, 137
MacLennan, K. *See* Hutchinson, H. B., *and* MacLennan, K.
Macquaire, P., Assay of the Amylolytic Diastase of Pancreas, 318
Major, F. *See* Parkes, A. E., *and* Major, F.
Malatesta, G. *See* Bianchi, A., *and* Malatesta, G.
Manning, R. J., *and* Merenstein, M., Note on Methoxyl Estimations, 134
Manzoff, C. D., Colour Reaction for the Detection of Methyl Alcohol, 264
Marc, R., *and* Sack, K., Estimation of Colloids in Effluents and the Use of the Interferometer in Water Analysis, 325
Marchadier, —, *and* Goujon, —, Poisonous Bran, 215
Marcus, J. K. *See* Curtman, L. J., *and* Marcus, J. K.
Marcusson, J., Analysis of the Asphalts, 404
————, Examination of Rosin Size, 139
Marden, J. W., Studies of Methods of Extraction by Means of Immiscible Solvents from Point of View of Distribution Coefficients. I.: Analysis of Vanilla Extracts and Extraction of Acetanilide, Saccharin, and Caffeine, 354
————. *See* Derby, I. H., *and* Marden, J. W.
Markus, F. *See* Mossler, G., *and* Markus, F.
Marqueyrol, —, *and* Muraour, —, Use of Diphenylbenzidine in Place of Diphenylamine as a Reagent for Oxidising Substances, 320
————, M. See Koehler, A., Marqueyrol, M., *and* Loriette, H.
Marriott, W. M., Estimation of Acetone, 178
————, Estimation of Small Quantities of Acetone in the Nephelometer, 178
————. *See also* Shaffer, P. A., *and* Marriott, W. M.
Marshall, E. K., jun., New Method for the Estimation of Urea in Blood, 39
————, F., Estimation of Potassium as Potassium Hydrogen Tartrate, 330

Masters, H., *and* Smith, H. L., The Changes in the Character of Fats during the Process of Cooking, 347
McCormack, H., Milling of Wheat and Testing of Flour, 358
McCrae, J., Abnormal Refraction of Milk Serum, 212
———, Ash of Silicotic Lungs, 359
McDermott, F. A., Use of the Concentrated Filament Tungsten Lamp in the Laboratory, 194
McFadden, A. W. J., Report on the Work of Inspectors of Foods for the Year 1912-1913, 195
McNab, L. *See* Jackson, L. C., McNab, L., *and* Rothera, A. C. H.
Mecklenburg, W., *and* Rosenkränzer, F., Colorimetric Estimation of Small Quantities of Hydrogen Sulphide, 325
Meerburg, P. A., Shrewsbury's Reaction for Paraffin, 43
Meillere, G., Estimation of Small Quantities of Lead, particularly in Urine in Cases of Lead Poisoning, 495
Meisl, E. *See* Klimont, J., *and* Meisl, E.
Meldrum, R., Determination of Melting-Points (of Fats, etc.) by the Thermometer Bulb Method, 61
Mellor, J. W., A Treatise on Quantitative Inorganic Analysis, with Special Reference to the Analysis of Clays, Silicates, and Related Minerals, *Review*, 155
Merenstein, M. *See* Manning, R. J., *and* Merenstein, M.
Mergen, W., *and* Winogradoff, A., Action of Halogens on Oleic Acid and Estimation of the Iodine Value of Fats, 311
Merl, T., "Vacuum Distillation" Method for the Detection of Formic Acid, 321
Merten, H. *See* Bömer, A., Krönig, R., Ley, R., *and* Merten, H.
Meserve, P. W. *See* Seidell, A., *and* Meserve, P. W.
Meyer, H., *and* Steiner, K., Estimation of Alkyl in Combination with Nitrogen, 226
———, J., Gravimetric Estimation of Selenium, 235
———, *and* Garn, W. von, Detection and Estimation of Very Small Quantities of Selenious Acid, 100
Meyerheim, G. *See* Holde, D., *and* Meyerheim, G.
Milbauer, J., *and* Pivnička, B., Analysis of Higher Oxides of Lead, 328
Milchsack, C., *and* Roth, W. A., Dennstedt Combustion Furnace with Internal Electrical Heating, 150
Miller, E. R., Taylor, G. W., *and* Eskew, M. H., Volatile Oil of *Calicanthus Floridus*, 558
———, J., Field Method for Determining Dissolved Oxygen in Water, 234
Misson, G., Colorimetric Estimation of Sulphur in Pig-Iron and Steels, 273
Mitchell, C. A., The Water of Dorton Spa, 210
———, J. P., Estimation of the Dust Fall in the Neighbourhood of Cement Plants, 372
Mitra, G. C. *See* Sutherland, W. D., *and* Mitra, G. C.
Molin, E., Method for Calculating Viscosity of Mineral Oil Mixtures, 446
Molinari, U., Reaction of Nitrates with Formic Acid, 233
Moore, R. *See* Desgrez, A., *and* Moore, R.
Moreau, L., *and* Vinet, E., Estimation of Traces of Arsenic, 230
Morgan, J. C., *and* James, C., Dimethyl Phosphates of the Rare Earths (Fractionation of Rare Earths), 146
Morton, H. A., Absorption of Dextrose by Bone-Black, 497
Mosca, F. T., Titanium and Rare Metals in Tobacco Ash, 43
Moser, L., *and* Schmid, O., Estimation of Carbon Monoxide by Means of Yellow Mercuric Oxide, 226
Mossler, G., *and* Markus, F., A Rapid and Simple Method for the Estimation of Alcohol in Tinctures, 393

Müller, E., *and* Seidel, F., Volumetric Estimation of Ferricyanide in Presence of Ferric Salts and Cyanides, 366
———, *and* Wegelin, G., Estimation of Periodate in Presence of Iodate and Iodide, 56
Iodimetric Estimation of Iodide Iodine, 99
-, U., Improved Absorption Tube for Spectroscopic Analysis, 153
Muraour, —. *See* Marqueyrol, —, *and* Muraour, —.
Murray, J. A., Steam Generator, 152

Nestler, A., Detection of Magnesium Sulphate in Saffron, 219
Neubauer, H., Estimation of Citric Acid Soluble Phosphoric Acid in Basic Slag by the Citrate Method and Lorenz's Method, 377
Neufeld, C. A., The Use of the Expressions "Cocoa Butter" and "Cocoanut Oil," 30
Nivière, J., Estimation of Esters in Essential Oils, 436
Njegovan, V., Estimating Extract in Wine, 221
Norton, A. B. *See* Gillett, H. W., *and* Norton, A. B.
Nottbohm, F. E., *and* Weinhausen, H., Estimation of Sulphate in Honey and other Substances Rich in Sugar, 259

Olson, G. A., Quantitative Estimation of Gliadin in Flour and Gluten, 33
———, Quantitative Estimation of the Proteins Soluble in Salt Solutions in Wheat Flour, 262
Opitz. H. *See* Klostermann, M., *and* Opitz, H.
Oppenheimer, M., Estimation of Lactic Acid in Liquids containing Protein, 133
O'Sullivan, J., The Chief Cause of the Loss of Sulphuric Anhydride and of Chlorine by Incinerating Substances Containing these Constituents, 425
Ottolenghi, D., Detection of Decomposition in Meats, 130

Pailler, E. C., Differentiation of Natural and Oil Asphalts, 362
Palkin, S. *See* Emery, W. O., *and* Palkin, S.
Palme, H., Electrolytic Method for Estimation of Mercury in Urine, 225
Palmenberg, O. W., Relation of Composition of Ash in Coal to its Fusing Temperature, 370
Palmer, L. S., *and* Wright, P. A., Ethyl Ester of Linolic Tetrabromide as a Product in the Analysis of Cottonseed Oil, 532
Paneth, F., *and* Hevesy, G. von, Radio-Elements as Indicators in Analytical Chemistry, 57
Parkes, A. E., *and* Major, F., The Composition and Analysis of Compound Liquorice Powder, 160
Paterson, J. H., *and* Forbester, R. E., Fractional Distillation of Mixed Pine Woods, 48
Paul, L., Melting-Points of Constituents of Colophony, 256
Paul, T., *and* Schantz, K., Apparatus for Determining Boiling-Points without Thermometer Corrections, 420
Pavesi, V., Aporeine and its Salts, 253
Payne, G. A. *See* Revis, C., *and* Payne, G. A.
Peche, K., Microchemical Detection of Myrosin, 409
———, New Tannin Reaction and its Relation to the Anthocyanenes, 412
Pellet, H., Determination of a Small Amount of Reducing Sugars in the Presence of a Large Proportion of Cane Sugar, 45
Pelt, G. Van, Investigation of the Methods of Analysis and Separation of Iron, Aluminium, Chromium, Zinc, and Manganese, 272
Perkins, A.E., Apparatus and Method for Determining the Hardness of Butter Fat, 282
Petersen, J., Detection of Antimony, 499

Petit, G. H., Detection of Traces of Aluminium, 142
Petkow, N., Composition and Adulteration of Rose Oil, 493
Petrie, J. M., Cyanogenetic Plants of New South Wales, 442
Pfanstiel, R. *See* Bedford, M. H., *and* Pfanstiel, R.
Pfyl, B., Volumetric Estimation of Phosphates in the Ash of Food Materials, 234
———, *and* Turnau, R., Volumetric Estimation of Casein in Milk by Means of the Tetraserum, 399
Philippe, E., Quantitative Sublimation, 194
Pick, H. *See* Auerbach, F., *and* Pick, H.
Pickard, J. A., *and* Potter, F. M., Oxygen Content of Open-Hearth Steel, 504
Piest, — Estimation of Sulphuric Acid in Nitrocelluloses, 45
Pieszczek, E., New Reaction for Boric Acid or Methyl Alcohol, 97
Pilcher, R. B., History of the Institute of Chemistry, 1877-1914, *Review*, 570
Pinkus, S. N., New Extraction Apparatus, 240
Pinoff, F., *and* Gude, K., Detection and Estimation of Lævulose in Presence of Other Sugars, 323
Pisani, F. New Reaction of Cocaine, 481
Pivnička, B. *See* Milbauer, J., *and* Pivnička, B.
Plaut, F., Absorption Phenomena in Abderhalden's Dialysis Process, 223
Poetschke, P., Sulphur Dioxide in Gelatin, 87
Pollard, C. T. *See* Turner, W. E. S., *and* Pollard, C. T.
Pontio, —, Direct Estimation of Caoutchouc in Crude Rubber, 179
————, Estimation of Caoutchouc in Crude Rubber, 138
Popp, M., Sources of Error in the Analysis of "Thomas" Meal, 377
Potmĕšil, R. *See* Votoček, E., *and* Potmĕšil, R.
Potter, F. M. *See* Pickard, J. A., *and* Potter, F. M.
Powell, C. W. R., Determination of the Viscosity of Refined Syrups and Molasses, 220
—————, Thermo-Regulator for Thermostats at Temperatures very close to Room Temperature, 568
Pozzi, W. *See* Siegfried, M., *and* Pozzi, W.
Pratt, W. R. *See* Corfield, C. E., *and* Pratt, W. R.
Prescott, J. A., Estimation of Phosphates in Soil Extracts, 329
———, S. C., *and* Winslow, C. A., Elements of Water Bacteriology, *Review*, 157
Prideaux, E. B. R., Titration of Boric Acid, 49
Pring, J. N., Estimation and Distinction of Ozone, Nitrogen Peroxide, and Hydrogen Peroxide at High Dilutions, 189
Prorsk, A. *See* Löb, W., *and* Prorsk, A.

Rabak, F., Aroma of Hops. Study of the Volatile Oil with Relation to the Geographical Sources of the Hops, 355
Rabaut, C. *See* Aloy, J., *and* Rabaut, C.
Radcliff, S., Extraction of Radium from Australian Ores, 455
Rae, W. N. *See* Joseph, A. E., *and* Rae, W. N.
Raikow, P. N., Action of Calcium on Acetone, 41
Rakshit, J. N., Estimation of Cane Sugar in the Presence of Lactose, 363
Rammstedt, O., Estimation of Acidity in Meal, Grist, and Bread, taking into Account the Action of Bacteria and Enzymes, 38
Rathgen, F., Microchemical Detection of Aluminium, 96
Ravizza, V., Rapid Testing of Potato Starch for Finishing Purposes, 94
Raynaud, F., Analyses of Ascitic Fluid, 38
Rea, F. W. *See* Letts, E. A., *and* Rea, F. W.
Reckert, F. C. *See* Gooch, F. A., Reckert, F. C., *and* Kuzirian, S. B.
Redman, L. V., Weith, A. J., *and* Brock, F. P., Determination of Phenol in the Presence of Hexamethylenetetramine and Formaldehyde, 266

Regester, S. H., Oxidation of Sulphur Compounds of Coal, and of Nitrogen in the Bomb Calorimeter, and the Correction to be Applied in Determining the Heating Value of Coal, 563
Reinau, E. *See* Klein, R., *and* Reinau, E.
Reutter, L., Analysis of Stabilised Cacao, 434
——————, Cacao Seeds, 171
Revis, C., *and* Payne, G. A., The Estimation of Cane-Sugar and Lactose in Condensed Milk by Means of Acid Mercuric Nitrate, 476
Richards, P. A. E., The Action of Weak Acids on Soluble Fluorides, 248
————, T. W., *and* Barry, F., Still for Exact Measurement of Boiling-Point during Fractional Distillation, 456
————————, *and* Shipley, J. W., Calibrating Thermometers by Means of Floating Equilibrium, 153
——————————————————, Freezing-Point of Benzene as a Fixed Point in Thermometry, 510
Richardson, B. P., Electro-Analytical Studies, 97
Riche, J. A., Improved Type of Calorimeter to be Used with any Calorimetric Bomb, 63
Richmond, H. D., The Composition of Milk and Milk Products, 243
Richter, E., Estimation of Berberine, 405
Riegler, E., Colorimetric Method for the Estimation of Albumin, 225
——————, Simple Colorimetric Method for the Estimation of Sugar in Urine, 224
Riffart, H. *See* Tillmans, J., Splittgerber, A., *and* Riffart, H.
Rinck, A., Detection of Methyl Alcohol, 407
Robertson, G. S., Solubility of Mineral Phosphates in Citric Acid, 146
Roettgen, J., Estimation of Lactic Acid in Wine, 37
Rosanoff, M. R., Bacon, C. W., *and* White, R. H., Rapid Laboratory Method of Measuring Partial Vapour Pressures of Liquid Mixtures, 569
Rose, J. K., Estimation of Zinc in Coinage Bronze by Volatilisation, 236
Roseman R., Influence of Ammonium Sulphate on the Rotation of Lactose, 133
Rosenheim, O., *and* Drummond, J. C., Volumetric Method for the Estimation of Ethereal and Inorganic Sulphates in Urine, 319
Rosenkränzer, F., Velocity of Solution of Blende and Galena in Dilute Sulphuric Acid, 452
————————. *See* Mecklenburg, W., *and* Rosenkränzer, F.
Rosenthaler, L., Detection of Hexamethylenetetramine in Wine or Milk, 125
Roth, W. A. *See* Milchsack, C., *and* Roth W. A.
Rothaug, G., Estimation of Chromium as Chromic Oxide, 97
Rothenfusser, S., Estimation of Glycerol in Wine, 87
Rothera, A. C. H. *See* Jackson, J. C., McNab, L., *and* Rothera, A. C. H.
Rozsenyi, I., Analysis of Coffee (Estimation of Roasted Cereals in Coffee Mixtures), 85
Rühle, J., Detection of Saponin, 175
Russo, C., New Method of Titrating Ferric Salts, 189
————, *and* Sensi, G., Reduction Method of Analysing Nitrates, Chlorates, and Peroxides, 188
Ryan, H., *and* Dillon, J., Montanin and Montana Waxes, 93

Sabatier, P., La Catalyse en Chimie Organique, *Review*, 65
Sack, K. *See* Marc, R., *and* Sack, K.
Sale, P. D. *See* Burgess G. K., *and* Sale, P. D.
Salomon, H., Estimation of the Unsaponifiable Constituents of Oils and Fats and of Lanolin by Means of the Digitonin Method, 310
Salvaterra, H., Volumetric Estimation of Nitro-Compounds and Nitroso-Compounds by Means of Titanium Chloride, 134

Sammet, O., Detection of Guaiacol in Urine, 360
Sander, A., Estimation of Free Sulphurous Acid in Presence of Thiosulphate or Sulphuric Acid, 279
———, Picric Acid as a Standardising Substance for Iodimetric and Acidimetric Titrations, 267
Sandman, E. A. *See* Thomas, J. B., *and* Sandman, E. A.
Sassa, R., Quantitative Estimation of the Oxyproteic Acid Fraction in Urine, 361
Scalione, C. C. *See* Burke, C. E., *and* Scalione, C. C.
Schaefer, G. L., Solvents for Alkaloids and Alkaloid Salts, 31
———, H. H. *See* Arny, H. V., *and* Schaefer, H. H.
Schanin, L., Melting-Point of Asphalt, 264
Schantz, K. *See* Paul, T., *and* Schantz, K.
Schellbach, H. *See* Hartwig, L., *and* Shellbach, H.
Schenk, K., Estimation of Total Sulphur in Commercial Benzene, 140
———, Rapid Analysis of Bronzes, 50
Schläpfer, P., Estimation of Water in Solid and Liquid Fuels by Distillation with Xylene, 141
Schlesinger, F. *See* Gutmann, S., *and* Schlesinger, F.
Schmid, O. *See* Moser, L., *and* Schmid, O.
Schmidt, E., Detection of Small Quantities of Selenious Acid in Sulphuric Acid, 419
Schmitz, W., Analysis of Golden Sulphide of Antimony, 186
Scholze, E. *See* Wolff, H., *and* Scholze, E.
Schönfeld, F., *and* Kunzel, E., Determination of Glycogen in Yeast, 129
Schorger, A. W., Leaf and Twig Oils of Cuban and Long-Leaf Pines and Cone Oil of Long-Leaf Pine, 498
———, Oils of the *Coniferæ.* Leaf and Twig, and Bark Oils of White Fir, 558
———, Study of Authentic Samples of Gum Turpentine, 436
Schowalter, E., Marshall's Manganese Reaction, 274
Schreiber, E., Estimation of Cholesterol and Oxycholesterol by the Method of Autenrieth and Funk, 129
Schumann, C. L. *See* Ware, E. E., *and* Schumann, C. L.
Schuppli, O. *See* Baragiola, W., *and* Schuppli, O.
Scott, J. M. *See* Snell, J. F., *and* Scott, J. M.
Sebelien, J., Beeswax of the Viking Period, 40
Seibert, F. M. *See* Burrell, G. A., *and* Seibert, F. M.
Seibriger, R., Estimation of Hop Bitter Substances by Cold Extraction with Carbon Tetrachloride, 125
Seidel, F. *See* Müller, E., *and* Seidel, F.
Seidell, A., *and* Meserve, P. W., Estimation of Minute Amounts of Sulphur Dioxide in Air, 376
Seidenberg, A., Detection of Gelatin in Sour Cream, 33
Sénéchal, A. *See* Bourion, F., *and* Sénéchal, A.
Senft, E., Microchemical Reactions of Certain Anthraquinones, 403
Sensi, G. *See* Russo, C., *and* Sensi, G.
Serger, H., Estimation of Salicylic Acid in Jams, 219
Sernagiotto, E., Apparatus for the Combustion of very Volatile Liquids, 566
Seyewetz, A. *See* Hanriot, M., Carré, P., Seyewetz, A., Charabot, E., *and* Hébert, A.
Shaffer, P. A., *and* Marriott, W. M., Estimation of Hydroxybutyric Acid, 184
Sharples, P. P., Relation between the Melting-Point and Viscosity of Refined Tars, 369
Shelley, F. F., Standardisation of Dried *Carica Papaya* Juice, 170
Shipley, J. W. *See* Richards, J. W., *and* Shipley, J. W.

Shrewsbury, H. S., Note on "Shrewsbury's Reaction for Paraffin," 296
———— ————, Two Rapid Methods of Estimating Water in Crude Petroleum, Oil-Fuel, and Similar Substances, 529
Siegfried, M., *and* Pozzi, W., Estimation of Small Quantities of Lead, 274
Sieveking, H. *See* Engler, C., Sieveking, H., *and* Koenig, A.
Sieverts, A., *and* Wippelmann, W., Rapid Electrolytic Separation of Copper from Arsenic, 371
Silvestri, G., Composition of "Dead" Rice, 550
————, Estimation of the Proteins in Wheat Flour, 550
Simmonds, C., The Estimation of Strychnine in the Presence of Quinine, 81
Sindall, R. W., *and* Bacon, W., The Examination of Commercial Gelatins with Regard to their Suitability for Paper-Making, 20
Sinnatt, F. S., *and* Cramer, B. J., The Determination of Carbon Monoxide in Air, 163
Sisley, P., *and* Freese, —, Diazonitro Reaction of Vegetable Oils, 228
Skarzynski, V. *See* Gauvin, R., *and* Skarzynski, V.
Slawik, P., Estimation of Cobalt in Steel, 271
Slyke, D. D. van, Estimation of Amino-Nitrogen in Organic Tissues, 175
——————, Gas-Volumetric Estimation of Free and Combined Amino-Nitrogen in Urine, 175
——————. *See* Levene, P. A., *and* Slyke, D. D. van
Smart, B. J., Some Observations on the Abel Heat Test, 44
Smetham, A., Valuation of Feeding-Stuffs by Means of Chemical Analysis, 481
Smit, J., Estimation of Mannitol, 93
———, Methods for the Estimation of Mannitol, 407
Smith, G. M. *See* Ball, T. R., *and* Smith, G. M.
——, H. G. *See* Baker, R. T., *and* Smith, H. G.
——, H. L. *See* Masters, H., *and* Smith, H. L.
——, O. F. *See* Wright, J. R., *and* Smith, O. F.
——, T. O., *and* James, C., Errors in the Estimation of Rare Earths as Hydroxides, 330
——, W. H, *See* Tuttle, J. B., *and* Smith, W. H.
Snell, J. F., *and* Scott, J. M., Analysis of Maple Products, II., 85.
Snelling, W. O., *and* Storm, C. G., Examination of Black Powder, 267
Sondén, K. *See* Barthel, C., *and* Sondén, K.
Spallino, R., Barium and Arsenic in Italian Tobacco, 43
————, Estimation of Nicotine as Silicotungstate, 43
————, Estimation of Nicotine in the Presence of Ammonia, 42
Spiers, C. W., Estimation of Tannin in Cider, 127
Splittgerber, A. *See* Tillmans, J., Splittgerber, A., *and* Riffart, H.
Sprinkmeyer, H., *and* Diedrichs, A., Detection of Beef and Mutton Fats in Lard by Bömer's Method, 253
Ssobolew, N. *See* Weisz, M., *and* Ssobolew, N.
Stadler, H. *See* Heubner, W., *and* Stadler, H.
Stanĕk, V., Determination of Cane Sugar in Beet Molasses by a Modification of the Double Polarisation (Clerget-Herzfeld) Method, using Citric Acid, 394
————, Muta-Rotation as a Source of Error in the Double Polarisation (Clerget-Herzfeld) Method of Determining Cane Sugar, 258
Stanford, R. V., Indigo-Forming Substances in Urine, 39
Steimmig, G., Note on Synthetic Caoutchouc from Isoprene, 179
Steiner, K. *See* Meyer, H., *and* Steiner, K.
Steuerwald, L. G. L., Constants for the Double Polarisation (Clerget-Herzfeld) Method of Determining Cane Sugar, 86
Stevens, H. P., The Estimation of Sulphur in Rubber, 74

Stevens, H. P. *See* Beadle, C., *and* Stevens, H. P.
Stiegler, H., Method for the Estimation of Crude Fibre, 367
Stiepel, C., Lathering Capacity of Soap, 312
———. *See* Leiste, —, *and* Stiepel, C.
Stokes, G. A., A Simple Fat Extractor, 295
Storm, C. G. *See* Snelling, W. O, *and* Storm, C. G.
Straszner, H. *See* Griesbach, W., *and* Straszner, H.
Strevens, J. L., Viscosity of Oils, 185
Strong, W. W., Instantaneous Thermostat and Smoke and Fume Monitors, Precipitators, and Recorders, 567
Strzyzowski, C., Centrifugal Method for Estimating Proteins in Urine, 40
Stüber, W. *See* Fendler, G., *and* Stüber, W.
Stutzer, A., Estimation of Nitrogen in Mixtures of Calcium Nitrate and Calcium Cyanamide, 329
Stüwe, W., Volumetric Estimation of Mercuric Chloride, 233
Suchtelen, F. H. H. van, *and* Itano, A., Electro-Titrimetric Method and its Application to General Analytical Chemistry, 565
Sugiura, K. *See* Falk, K. G., *and* Sugiura, K.
Suleimann, A. *See* Hunder, M., *and* Suleimann, A.
Sutcliffe, J. A. L., Note on the Insoluble Bromide Value of Oils, 388
———, Notes on the Insoluble Bromide Test for Drying Oils, 28
Sutherland, W. D., *and* Mitra, G. C., Modification of Teichmann's Test for Blood, 315
Swaving, A. J., Recueil de Législations Beurrières et Margarinières Concernant la Répression des Fraudes dans le Commerce du Beurre, *Review*, 474
Symons, C. T., Modification of Teichmann's Test for Blood, 128
———. *See* Browning, K. C., *and* Symons, C. T.

Tanberg, A. P., Pyridine as a Solvent in the Estimation of Hydroxyl Groups by Means of Alkyl Magnesium Halides, 186
Tanret, C., Variety of Starches, 314
Tatlock, R. R., *and* Thomson, R. T., On Damage Caused to Vegetation by Sulphurous and Sulphuric Acids in the Atmosphere, 203
Taylor, G. B., Apparatus for Analysis of Complex Gas Mixtures, 566
———, G. W. *See* Miller, E. R., Taylor, G. W., *and* Eskew, M. H.
Thatcher, R. W., *and* Koch, G. P., Quantitative Extraction of Diastases from Plant Tissues, 260
Thiel, A., Properties of the Phthaleïns as Indicators, 136
Thies, F. H., Estimation of Carbon by the Wet Way. Simultaneous Estimation of Halogens, 132
Thomas, J. B., *and* Sandman, E. A., Results of the Hypochlorite Disinfection of Water Supplies. A Comparison of the Reduction of the Different Members of the *Bacillus Coli* Group, 443
Thompson, W. H., Wallace, T. A., *and* Clotworthy, H. G. S., Folin Method for the Estimation of Creatine and Creatinine, 41
Thomson, R. T., Methyl Red as an Indicator, 518
———, Notes on Flour: 1. Acidity of Flour; 2. Natural and Artificial Bleaching of Flour; 3. Sulphates and Lime in Flour, 519
———, *See* Tatlock, R. R., and Thomson, R. T.
Thornton, W. M., jun., Use of Ammonium Salt of Nitrosophenylhydroxylamine (Cupferron) in Quantitative Separation of Titanium from Iron, 332
———, *and* Hayden, E. M., jun., Separation of Zirconium from Iron and Aluminium with the Aid of the Ammonium Salt of Nitrosophenylhydroxylamine ("Cupferron"), 565

Thüringer, V. *See* Wunder, M., *and* Thüringer, V.
Tillmans, J., Water Purification and Sewage Disposal, *Review*, 66
————, Splittgerber, A., *and* Riffart, H., Estimation and Significance of Ammonia in Milk, 173
——————————————, Preservation of Milk Samples for Analysis, 398
Tolman, L. M., *and* Goodnow, E. H., Composition of Cider Vinegars Made by Generator Process, 36
Toninelli, A., Specific Reaction of Ethyl Alcohol, 319
Torossian, G., Efficient Boiling-Rod, 237
Tortelli, M., *and* Ceccherelli, A., Estimation of Glycerol in Technical Glycerines and Soap Lyes, 181
Trickey, J. P. *See* Lee, R. E., Trickey, J. P., *and* Fegely, W. H.
Trümpler, G. *See* Baur, E., *and* Trümpler, G.
Trutzer, E., Filter from which a Precipitate may be Removed Quantitatively, 151
Tschermak, G., Estimation of Silicic Acids, 455
Tschirwinsky, P., Quantitative Mineralogical and Chemical Composition of the Granites and Gneisses, 98
Tschugaeff, L., *and* Chlopin, W., New Method for Determining Solubilities at High Temperatures, 335
Turnau, R. *See* Pfyl, B., *and* Turnau, R.
Turner, W. E. S., *and* Pollard, C. T., Improved Apparatus for the Determination of Molecular Weight by the Landsberger-Sakurai Method, 152
Tutin, F., Constituents of Senna Leaves, 35
Tuttle, J. B., *and* Smith, W. H., Printing Inks and their Analysis, 450
Twining, R. H. *See* White, G. F., *and* Twining, R. H.
Twitchell, E., Melting and Solidifying Points of Mixtures of Fatty Acids and Use of these Points to Determine Composition of Such Mixtures, 448

Utt, C. A. A., Earth-Nut Butter, 481
Utz, —, Detection of Carbon Disulphide in Extracted Oils, 180
————, Estimation of Total Sulphur in India-Rubber, 368
————, Modification of Halphen's Test for Cotton-Seed Oil, 92

Van Eck, J. J., Estimation of Dissolved Oxygen in Water, 55
Van Pelt, G., Investigation of the Methods of Analysis and Separation of Iron, Aluminium, Chromium, Zinc, and Manganese, 272
Vaubel, W., and Weinerth, E., Comparative Examinations of Raw Caoutchouc and Caoutchouc by Bromination Methods, 226
Verbeek, P., New Differential Pressure Gauge, 60
————, Two New Colorimeters, 282
Verda, A., Detection of Adulterants in Saffron by Means of Phospho-molybdic Acid, 219
Vignon, L., Action of Solvents on Coal, 406
————, Solvents for Coal, 320
Villiers, A., Manganese Sulphide and the Estimation of Manganese, 417
Vilmorin, P. de, *and* Levallois, F., The Hydrolysis of Lævulosans and its Application in Plant Analysis, II. and III., 88
Vinet, E. *See* Moreau, L., *and* Vinet, E.
Vinograd, M., Estimation of Arsenic in Organic Substances, 442
Vivario, R., Detection of Methyl Alcohol in Spirits, 433
Voorhees, S. S. *See* Walker, P. H., *and* Voorhees, S. S.
Voswinckel, H., Apparatus for Catalytic Reductions with Hydrogen, 333
Votoček, E., *and* Potměšil, R., Estimation of Rhamnose in Presence of Other Methylpentoses, 410

Vournasos, A. C., Double-Walled Crucible for Ignitions, 282
Vuk, M., Nickel Cooking-Vessels, 417

Waele, A. de, The Fachini-Dorta Method of [Approximately] Separating Liquid from Solid Fatty Acids in Oils and Fats, 389
Wagenaar, M., Microchemical Reaction of Caffeine, Theobromine, Theophylline, and their Derivatives with Mercuric Chloride, 217
Wales, H. E., Specific Heat of California Petroleums, 499
Walker, A. W. *See* Kendall, A. I., Day, A. A., *and* Walker, A. W.
———, P. H., *and* Voorhees, S. S., Tests of Paints for Steel, Subjected to Alternate Exposure to Air and Fresh Water, 54
Wallace, T. A. *See* Thompson, W. H., Wallace, T. A., *and* Clotworthy, H. G. S.
Waltuch, R. *See* Zerner, E., *and* Waltuch, R.
Ware, E. E., *and* Schumann, C. L., Examination of China Wood Oil, 557
Warth, F. J., *and* Darabsett, D. B., Fractional Liquefaction of Rice Starch, 440
Waterman, N., Differentiation between Hen and Duck Egg Albumins for Commercial Purposes, 222
Wedemann, W., Schardinger's Formaldehyde-Methylene Blue Reaction and Other Ferment Reactions of Goat's Milk, 218
Wegelin, G., Volumetric Estimation of Vanadium, 148
———. *See* Müller, E., *and* Wegelin, G.
Weigmann, —, *and* Haglund, E., Estimation of Water in Cheese by Mai and Rheinberger's Method, 171
Weinerth, E. *See* Vaubel, W., *and* Weinerth, E.
Weinhausen, H. *See* Nottbohm, F. E., *and* Weinhausen, H.
Weise, G. L. *See* Gutbier, A., *and* Weise, G. L.
Weiser, S., *and* Donath, H. C., Comparison of Various Methods for Determining the Iodine Value of Fats, 406
Weiss, J. M., Estimation of So-called Free Carbon in Tar Products, 364
Weisz, M., *and* Ssobolew, N., Colorimetric Estimation of Histidine, 92
Weith, A. J. *See* Redman, L. V., Brock, F. P., *and* Weith, A. J.
Wempe, G., Gas Analysis Apparatus, 421
Wenger, P. *See* Wunder, M., *and* Wenger, P.
Wesson, L. G., Combustion Method for the Direct Estimation of Rubber, 368
West, A. P., Analysis and Composition of Red Lead, 375
Wewerinke, J. *See* Fischer, K., *and* Wewerinke, J.
Weyman, G., Difference between Calculated and Determined Calorific Values of Coal Gas, 132
Whipple, G. C., The Microscopy of Drinking-Water, *Review*, 472
White, G. F., *and* Twining, R. H., New Viscosimeter, 155
———, R. H. *See* Rosanoff, M. R., Bacon, C. W., *and* White, R. H.
Wiesner, G. H., Nitrogen and Chlorine in Rain and Snow, 190
Wiley, H. W., Principles and Practice of Agricultural Analysis, *Review*, 423
Wilson, A. C., Improved Apparatus for the Analysis of Mixed Gases, 391
Windisch, R., Refraction of Milk Serum (Ackermann's Method), 256
Winkler, L. W., Estimation of Hardness in Water, 379
Winmill, T. F. *See* Graham, J. I., *and* Winmill, T. F.
Winogradoff, A. *See* Mergen, W., *and* Winogradoff, A.
Winslow, C. A. *See* Prescott, S. C., *and* Winslow, C. A.
Wippelmann, W. *See* Sieverts, A., *and* Wippelmann, W.
Wlokka, A. *See* Bode, G., *and* Wlokka, A.
Wohack, F., Detection of Coal-Tar Dyes in Wines, 358
Woker, G., *and* Belencki, E., Influence of Metals Present in Urine on the Velocity of the Reduction Tests for Dextrose, 131

Wolff, H., Examination of Bleached Shellac, 139 and 411
———, *and* Scholze, E., Estimation of Rosin in Varnishes, Oils, and Soaps, 228
Woodcock, W. H., *and* Blount, B., Sulphuretted Hydrogen from Artificial Graphite, 67
Wrenshall, R. *See* Jamieson, G. S., *and* Wrenshall, R.
Wright, A. G. *See* Cripps, R. A., *and* Wright, A. G.
Wright, J. R., *and* Smith, O. F., Quantitative Determination of Radium Emanation in the Atmosphere, and its Variation with Altitude and Meteorological Conditions, 418
Wright, P. A. *See* Palmer, L. S., *and* Wright, P. A.
Wunder, M., *and* Thüringer, V., Estimation of Palladium by Means of α-Nitroso-β-Naphthol and Separation of Palladium from Copper and Iron, 55
———, *and* Wenger, P., Separation of Beryllium from Uranium, 325
Wüstenfeld, H., *and* Foehr, C., Pycnometer Spindle, a New Instrument for Use in the Estimation of Alcohol, 284

Yanagawa, H., Violet Sodium-Nitro-Prusside Reaction of Urine, 264

Zaleski, L., Volumetric Estimation of Small Quantities of Potash, 417
Zerewitinoff, J., Estimation of Hydroxy Fatty Acids in Fats by Means of Magnesium Methyl Iodide, 41
Zerner, E., *and* Waltuch, R., Sugars in Urine in Cases of Pentosuria, 40
Zuccàri, G., Arsenic as a Normal Constituent in Soils, 49
Zwikker, J. J. L., Apparatus for Ether Extraction of Liquids, 239

INDEX OF SUBJECTS.

ABDERHALDEN'S Dialysis Process, Adsorption Phenomena in, 223
Abel Heat Test, Observations on the, Some, 44
Absorption Tube, Spectroscopic Analysis, Improved, for, 153
ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS: 31, 84, 122, 171, 215, 252, 308, 352, 393, 433, 480, 532
Acetanilide and Phenacetin in Admixture, Estimation of, 433
(————, Vanilla Extracts, I. Analysis of, and Extraction of, Saccharin, and Caffeine.) Study of Methods of Extraction by Means of Immiscible Solvents from Point of View of Distribution Coefficients, 354
Acetone, Calcium, Action on, of, 41
————, Estimation of, 178
————, Nephelometer, Estimation of Small Quantities of, in the, 178
Acetyl Value, Oils, Fats, etc., Estimation of the, of, 362
Acid Mercuric Nitrate, Cane-Sugar and Lactose in Condensed Milk, The Estimation by Means of, of, 476
—— Radicals, Chlorides, Chlorates, Perchlorates, Bromides, Bromates, and Fluorides, the Estimation of the, in. Application of Sodium Paratungstate and the Blowpipe Flame to, 188
1. Acidity of Flour, 2. Natural and Artificial Bleaching of Flour, 3. Sulphates and Lime in Flour. Notes on Flour, 519
————, Meal, Grist, and Bread, Estimation of, in, Taking into Account the Action of Bacteria and Enzymes, 38
Acids, Soluble Fluorides, The Action of Weak, on, 248
Adiabatic Calorimeter, 281
Adsorption Phenomena in Abderhalden's Dialysis Process, 223
Adulterants, Saffron, Detection of, in, by Means of Phosphomolybdic Acid, 219
Aerated Waters, Saponin, Detection in, of, 440
Aeration, De-Aerated Liquids, New Seal for Prevention of, in, 509
Africa, Copals from British West, 396
Air Analysis, Application to: Analysis of Very Small Quantities of Gas, 413
——, Carbon Dioxide, Estimation in, of, 324
——, Carbon Monoxide, The Determination of, in, 163
——, Dust and Bacteria Samples of, Apparatus for Taking, 283
——, Paints for Steel Subjected to Alternate Exposure to, and Fresh Water, Tests for, 54
——, Sulphur Dioxide in, Estimation of Minute Amounts of, 376
d-Alanine, *d*-Valine, Separation of, and, 178
Albumin, Colorimetric Method for the Estimation of, 225
Albumins, Hen and Duck Egg, Differentiation between, for Commercial Purposes, 222
Alcohol, Boric Acid or Methyl, New Reaction for, 97
, Ethyl, and Methyl, Detection of, 184
Methyl, and Ethyl, Detection of, 184
———, Colour Reaction for the Detection of, 264
———, Detection of, 407

Alcohol, Methyl, in Corn Silage, Occurrence of, 549
———, Milk, Titration with, at Different Degrees of Concentration of, 317
———, Pycnometer Spindle, a New Instrument for Use in the Estimation of, 284
———, Spirits, Detection of Methyl, in, 433
———, Tinctures, Rapid and Simple Method for the Estimation of, in, 393
———, Tropics, Estimation in the, 480
Alcoholometry, Notes on, II., 532
Aldoses, Estimation of (Part I.). Action of Neutral Sodium Hypoiodite, 556
Alfalfa, Enzymes Present in, (Lucerne), 552
Aliphatic Substances, Wet Way, Volumetric Estimation of Carbon in, in the, 556
Alkali Bromate, Ferrocyanide, Estimation by Titration with, of, 366
—— Carbonate Solutions, Lead Carbonate, Basic Lead Carbonate, and Lead Sulphate, Behaviour towards, of, 53
—— Carbonates, Hydrochloric Acid, Convenient Standard End-Point in Titration of, with, 268
——, Commercial Soda and Soaps, Estimation of Free, in, by the Barium Chloride Method, 229
—— Cyanides, Hydrocyanic Acid and, New Method for Determination of, 448
——, Soaps, Estimation of Free, in, 140
Alkaline Solution, Oxidation in, Estimation of Chromium by, 143
Alkaloid Salts, Solvents for Alkaloids and, 31
Alkaloids, Estimation of the, Application to Preparations of Nux Vomica, 122
————, Solvents for, and Alkaloid Salts, 31
Alkyl Magnesium Halides, Hydroxyl Groups, Pyridine as a Solvent in the Estimation of, by Means of, 186
——, Nitrogen, Estimation of, in Combination with, 226
Allantoin, Urine, Estimation of, in, 263
Alloys, Approximate Melting-Points of Some Commercial Copper, 98
——, Cerium and Iron, Analysis of Pyrophoric, of, 415
——, Copper, Tin, and Silicon, Analysis of, 500
——, The Determination in Platinum-Iridium, of Iridium, 340
——, Nomenclature, Summary of First Report of the Committe on the, of, *Report*, 286
Aluminium, Fluorides, Precipitation of, in Presence of, 269
————, Iron, Chromium, Zinc, and Manganese, Investigation of the Methods of Analysis and Separation of, 272
————, Iron and, Separation of Zirconium from, with the Aid of the Ammonium Salt of Nitrosophenylhydroxylamine ("Cupferron"), 565
————, Microchemical Detection of, 96, 269
————, Presence of Gallium in Commercial, 53
————, Silica, Iron, and, in Mineral Waters, Estimation of, Formation of Sediment in Bottled Mineral Waters, 126
————, Traces of, Detection of, 142
Amino-Acid, Barley, Malt, and Beer, Estimation of, and Polypeptide-Nitrogen in, by Titration with Acid after Treatment with Formaldehyde, 257
Amino-Acids, Blood, Estimation of, in, 315
—————, Proteins and, Herzig and Meyer's Reaction Applied to, 317
Amino-Nitrogen, Organic Tissues, Estimation of, in, 175
——————, Urine, Gas-Volumetric Estimation of Free and Combined, in, 175
Ammonia, Illuminating Gas, Estimation of, in, 369
————, Milk, Estimation and Significance of, in, 173
————, Nicotine in the Presence of, Estimation of, 42
Ammonium Molybdate, Lead, Effect of Tungsten on, Assay for, 192
———— Salt, Nitrosophenylhydroxylamine ("Cupferron"), Separation of Zirconium from Iron and Aluminium with the Aid of the, of, 565

Ammonium, Salt, Nitrosophenylhydroxylamine ("Cupferron"), Use of, in Quantitative Separation of Titanium from Iron, 332
——— Sulphate, Lactose, Influence of, on the Rotation of, 133
Amylolytic Diastase, Pancreas, Assay of the, of, 318
Analyses, Wine, Method of Expressing Results of, According to the Latest Chemico-Physical Principles, 127
Analysis, Chemical, The Properties of Some Chlorhydrocarbons and their Uses in, 4, 385
———, Nephelometer, New, and its Use in, 283
Analytical Characters, Benzoin, of, 393
——— Chemistry, General, Electro-Titrimetric Method and its Application to, 565
———————, Investigation Scheme, 337
———————, Radio-Elements as Indicators in, 57
——— Procedure, Thirty per cent. Hevea Rubber Insulating Compound, for, 137
Animal Fats, Phytosterol in, Detection of, 84
———, Vegetable Fats in, Detection of, 32
———, Unsaponifiable Constituents of Mowrah Fat and the Detection of this Fat in, and Vegetable, 310
Anise, Essential Oils and Essential Oil Products, Hydrogen Value of Some. I.: Oils of Sassafras, Fennel, Clove, and Pimento, 557
Annual Address, Retiring President, of the, 108
Anode, Rotating Cathode and, for Rapid Electrolytic Analysis, 334
Anthocyanenes, Tannin Reaction, New, and its Relation to the, 412
Anthraquinones, Microchemical Reactions of Certain, 403
Antimony, Detection of, 499
———, Golden Sulphide of, Analysis of, 186
Antipyrin, Estimation of, 480
Aporeine, Salts, and its, 253
APPARATUS, ETC., ABSTRACTS: 59, 103, 149, 193, 237, 280, 333, 381, 420, 456, 508, 565
Arabinose, Pentoses, Xylose and, Methods of Estimation of Carbohydrates. III. The Cupric-Reducing Power of the, 555
Arachidic Acid, Fatty Acids, Note on the, and Detection of, 122
Aroma, Hops, of. Study of the Volatile Oil with Relation to the Geographical Sources of the Hops, 355
Aromatic, Volatile Oils in, Plants, Drugs, and Spices, Estimation of, by the Tail-ameter, 35
Arsenic, Copper and Iron, Iodimetry of, 116
———, ———, Rapid Electrolytic Separation from, of, 371
———, Hydrochloric and Sulphuric Acids, Estimation of, 370
———, Hypophosphorous Acid, Quantitative Separation of, from the Metals by Means of, 269
———, Italian Tobacco, Barium and, in, 43
———, Organic Substances, Estimation of, in, 442
———, Soils, as a Normal Constituent in, 49
———, Traces of, Estimation of, 230
Arsenite Solutions, Standardisation of Volumetric, 271
Ascitic Fluid, Analyses of, 38
Ash, Coal, Relation of Composition of, in, to its Fusing Temperature, 370, 445
——, Lungs, of Silicotic, 359
——, Phosphates in the, of Food Materials, Volumetric Estimation of, 234
——, Tobacco, Titanium and Rare Metals in, 43
Asphalt, Melting-Point of, 264

Asphaltic Matters, Dark Mineral Oils, Estimation of, in, 178
Asphalts, Analysis of, 131
——————— the, 404
————, Natural and Oil, Differentiation of, 362
Atmosphere, Radium Emanation in the, Quantitative Determination of, and its Variation with Altitude and Meteorological Conditions, 418
Atomic Weights, International, 1914, 3
Australian Eucalypts, Tasmanian and, Correlation between the Specific Characters of the, 435
———— Ores, Radium, Extraction from, of, 455
Autenrieth and Funk, Cholesterol and Oxycholesterol, Estimation of, by the Method of, 129
Automatic Gas-Sampling Apparatus, with some Observations on Sampling, 103

Bacillus Coli Group, Water Supplies, Results of the Hypochlorite Disinfection of, A Comparison of the Reduction of the Different Members of the, 443
Bacteria, Acidity in Meal, Grist, and Bread, Estimation of, Taking into Account the Action of, and Enzymes, 38
————, Dust and, Samples of Air, Apparatus for Taking, 283
Bacterial Metabolism in Milk, Studies in, 494
BACTERIOLOGICAL, PHYSIOLOGICAL, ETC., ABSTRACTS: 38, 88, 128, 175, 222, 260, 315, 359, 402, 442, 494, 551
Balsam, Peru, 400
Barium Carbonate Titration Method, Steel and Iron, Determination by the, of Carbon in, 373
———— Chloride, Commercial Soda and Soaps, Estimation of Free Alkali in, by the, Method, 229
——————— Method, Moslinger's, Estimation of Lactic Acid in Wine by, 397
———— and Arsenic in Italian Tobacco, 43
Bark Oil, Oils of the *Coniferæ*, Leaf and Twig, and, of White Fir, 558
Barley, Amino-Acid and Polypeptide-Nitrogen in, Malt, and Beer, Estimation of, by Titration with Acid after Treatment with Formaldehyde, 257
Base Metal, Precious Metal Bullion, Determination of Silver and, in, 502
Basic Lead Carbonate, Alkali Carbonate Solutions, Behaviour of Lead Carbonate, and Lead Sulphate towards, 53
——— Slag, Citrate Method and Lorenz's Method, Estimation of Citric Acid Soluble Phosphoric Acid in, by the, 377
—————, Phosphoric Acid in, Estimation of Citric Acid Soluble, 377
Beans, Cacao, Composition of the Radicles of, 308
Beef and Mutton Fats, Lard, Detection by Bömer's Method of, in, 253
——— -Fat, Lard, Detection of, in, 84, 171
———————, Detection by Polenske's and Bömer's Methods of, in, 216
Beer, Amino-Acid and Polypeptide-Nitrogen in Barley, Malt, and, Estimation of, by Titration with Acid after Treatment with Formaldehyde, 257
Beers, Electrical Conductivity of, 352
———, Specific Gravities of Malt Worts and, Temperature Corrections to be Applied to the, 256
Beeswax, Commercial Analysis of, 123
————, Viking Period, of the, 40
Beet Molasses, Cane Sugar in, Determination of, by a Modification of the Double Polarisation (Clerget-Herzfeld) Method, Using Citric Acid, 394
——— Products, Double Polarisation Process, Comparative Study of Different Methods of Determining Cane-Sugar in, by the, 435

Beet Products, Raw Cane Products, Detection of Raw, in Admixture with, 258
Benzene, Freezing-Point of, as a Fixed Point in Thermometry, 510
———, Total Sulphur in Commercial, Estimation of, 140
Benzoin, Analytical Characters of, 393
Benzoyl, Derivatives, Estimation of, in, 405
Berberine, Estimation of, 405
Bernadot's Vinometer, 457
Beryllium, Separation and Estimation of, 232
———, Uranium, Separation of, from, 325
Biological and Clinical Problems, Dialysis Method, Utility of, in, 223
Black Powder, Examination of, 267
Black Sage, Oil of, Investigations on, 559
Blast Furnace, Portland Cement, Estimation of, Slag in, 187
Bleached Shellac, Examination of, 139, 411
Bleaching, Flour, Notes on, 1. Acidity of Flour; 2. Natural and Artificial, of Flour; 3. Sulphates and Lime in Flour, 519
Blende, Dilute Sulphuric Acid, Velocity of Solution of, and Galena in, 452
Blood, Amino-Acids, Estimation in, of, 315
———, and Other Physiological Liquids, Carbohydrates and Lactic Acid in, Estimation of, 551
———, Hæmatin in, Estimation of, 262
——— Serum, Chlorine in, Estimation of, 223
——— -Serum, Lipoids in, Estimation of, 176
———, Sugar in, Estimation of, 90
———, Sugar in, Micro-Estimation of, 90
———, Teichmann's Test for, Modification of, 128
———————————, 315
———, Urea in, New Method for the Estimation of, 39
Blowpipe Flame, Acid Radicles in Chlorides, Chlorates, Perchlorates, Bromides, and Bromates, Fluorides, Application of Sodium Paratungstate and the, to the Estimation of the, 188
Body, Varnish, Quantitative Determination of, and Solvent in, 560
Bog Iron Ore, Organic Matter in, Destruction of, 372
Boiling-Point, Still for Exact Measurement of, during Fractional Distillation, 456
———-Points, Thermometer Corrections, Apparatus for Determining, without, 420
———-Rod, Efficient, 237
Bolting, Flour, Estimation of "Cellulose" in, as a Measure of the Degree of, 309
Bomb Calorimeter, Coal, Oxidation of Sulphur Compounds of, and of Nitrogen in the, and the Correction to be Applied in Determining the Heating Value of Coal, 563
Bomb, Improved Type of Calorimeter to be Used with any Calorimetric, 63
Bömer's and Polenske's Methods, Beef Fat in Lard, Detection by, of, 216
——— Method, Lard, Detection of Beef and Mutton Fats by, in, 253
——— Phytosterol Acetate Test, Butter Fat, Detection of Coconut Oil in, by Means of, and Polenske's Distillation Method, 254
Bone-Black, Dextrose, Absorption by, of, 497
Boric Acid, Estimation of, in, 123
——— in Organic Substances, Method of Estimating Very Small Quantities of, 96
———, Methyl Alcohol, New Reaction for, or, 97
———, Milk and Eggs, Occurrence of, in, 123
———, Titration of, 49
Bottled Mineral Waters, Formation of Sediment in, Estimation of Silica, Iron, and Aluminium in Mineral Waters, 126
Bran, Poisonous, 215

Brass, Bronze and, Method for Rapid Quantitative Analysis of, 453
Bread, Acidity in Meal, Grist, and, Estimation of, Taking into Account the Action of Bacteria and Enzymes, 38
British West Africa, Copals from, 396
Bromates, Acid Radicals in Chlorides, Chlorates, Perchlorates, Bromides, and Fluorides, Application of Sodium Paratungstate and the Blowpipe Flame to the Estimation of the, 188
Bromides, Acid Radicals in Chlorides, Chlorates, Perchlorates, Bromates, and Fluorides, Application of Sodium Paratungstate and the Blowpipe Flame to the Estimation of the, 188
———, Thiocyanates and Ferrocyanides, Detection of, in Presence of, Action of Chromic Acid on Cyanogen Bromide, 412
———, Thiocyanates and Ferrocyanides, Detection of, in Presence of, 187
Bromination Methods, Raw Caoutchouc and Caoutchouc, Comparative Examinations by, of, 226
Bromine, Gases and Vapours, Estimation of, in. VII. Communication, 271
———, Telluric Acid, Use of, in Estimation of, Associated with Chlorine in Haloid Salts, 331
———, Turpentine Oil, Thermal Value of, 314
Bronze and Brass, Method for Rapid Quantitative Analysis of, 453
———, Zinc in Coinage, Estimation of, by Volatilisation, 236
Bronzes, Rapid Analysis of, 50
Brucine, Strychnine, Estimation in Presence of, of, 394
Bulb Method, Melting-Points (of Fats, etc.), Determination of by the Thermometer, 61
Bullion, Precious Metal, Determination of Silver and Base Metal in, 502
Butter, Cacao, A Skilfully Adulterated, 216
———, Direct Estimation of, Combustion Method for the, 368
———, Earth-Nut, 481
——— Fat, Coconut Oil in, Detection of, by Means of Polenske's Distillation Method and Bömer's Phytosteryl Acetate Test, 254
———, Hardness of, Apparatus and Method for Determining the, 282
——— in Cheese, and Cream, Rapid Estimation of, 253
—— Margarine, Content of, 216
Substitute, Cacao, 434
Cacao Beans, Composition of the Radicles of, 308
——— Butter, A Skilfully Adulterated, 216
——— Substitute, 434
—— Seeds, 171
Stabilised, Analysis of, 434
Caffeine, Acetanilide, Saccharin, and. I. Extraction of, and Analysis of Vanilla Extracts, Study of Methods of Extraction by Means of Immiscible Solvents from Point of View of Distribution Coefficients, 354
———, Coffee, Estimation of, in, 394
———, Kola Preparations, Estimation of, in, 31
———, Mercuric Chloride, Microchemical Reaction of, Theobromine, Theophylline, and their Derivatives with, 217
Cakes, Cottonseed, and Meals, Estimation of Husks in, 173
Calcium, Acetone, Action of, on, 41
——— and Magnesium in the Same Solution, Titration of, 51
——— Cyanamide, Mixtures of Calcium Nitrate and, Estimation of Nitrogen in, 329
———, Magnesium, Separation of, from, 142
——— Nitrate, Nitrogen in Mixtures of, and Calcium Cyanamide, Estimation of, 329

Calcium, Oxalate, Gravimetric Estimation of, as, 50
Calibrating Thermometers by Means of Floating Equilibrium, 153
Calicanthus Floridus, Volatile Oil of, 558
California Petroleums, Specific Heat of, 499
Calorific Values, Coal Gas, Difference between Calculated and Determined, of, 132
Calorimeter, Adiabatic, 281
———, Calorimetric Bomb, Improved Type of, to be Used with any, 63
Camphor, Tablets and Pills, Estimation of, in, 353
Cane Products, Raw Beet Products, Detection in Admixture with Raw, of, 258
Cane Sugar, Acids in Water-Alcohol Solutions, Inversion of, by, 323
———, Beet Molasses, Determination of, in, by a Modification of the Double Polarisation (Clerget-Herzfeld) Method, Using Citric Acid, 394
———, Beet Products, Comparative Study of Different Methods of Determining, in, by the Double Polarisation Process, 435
———, Condensed Milk, The Estimation by Means of Acid Mercuric Nitrate of, and Lactose in, 476
———, Double Polarisation (Clerget-Herzfeld) Method of Determining, Constants for the, 86
———, Lactose, Estimation of, in the Presence of, 363
———, Muta-Rotation as a Source of Error in the Double Polarisation (Clerget-Herzfeld) Method of Determining, 258
———, Reducing Sugars, Determination of a Small Amount of, in the Presence of a large Proportion of, 45
Caoutchouc, Crude Rubber, Direct Estimation of, in, 179
———, ———, Estimation of, in, 138
———, Isoprene, Note on Synthetic, from, 179
———, Raw, and, Comparative Examinations of, by Bromination Methods, 226
——— Mixtures, Loading in, Estimation of, According to the Standards of the Verband Deutscher Elektrotechniker, 91
Capillary Lift, Soils, Measuring the, of, 318
Carbohydrates, Blood and Other Physiological Liquids, Estimation of, and Lactic Acid in, 551
———, Methods of Estimation of. III.: The Cupric-Reducing Power of the Pentoses—Xylose and Arabinose, 555
———, Picrate Colorimetric Method for the Estimation of, 180
Carbon, Aliphatic Substances, Volumetric Estimation of, in, in the Wet Way, 556
———, Barium Carbonate Titration Method, Determination of, in Steel and Iron by the, 373
——— Dioxide, Air, Estimation of, in, 324
———, and Plants, 260
———, Mineral Waters, the Approximate Estimation of Free, in, Apparatus for, 238
——— Disulphide, Extracted Oils, Detection of, in, 180
——— Monoxide, Air, The Determination of, in, 163
--, Estimation of, 445
---, Yellow Mercuric Oxide, Estimation of, by Means of, 226
, Soils and Soil Extracts, Estimation of, in, 449
, Steel, Determination by the Direct Combustion Method of, in, 560
, Tar Products, Estimation of So-Called Free, in, 364
Tetrachloride, Drugs and Medicinal Plants, Use of, in the Extraction and Estimation of the Active Principles of, 255
--, Hop Bitter Substances, Estimation of, by Cold Extraction with, 125
-, Wet Way, Estimation of, by the. Simultaneous Estimation of Halogens, 132

Carbonic Acid, Small Quantities of, Titration of, 143
Carica Papaya Juice, Dried, Standardisation of, 170
Casein, Ferric Ammonium Sulphate, Estimation of, by Means of, 496
——, Milk, Volumetric Estimation of, in, by Means of the Tetraserum, 399
Castor Bean, Urease, Experiments with (Studies on Enzyme Action, XI.), 553
—— Seeds, Detection of, The, 292
Catalytic Reductions, Hydrogen, Apparatus for, with, 333
Cathode, Electrolysis with the Rotating, Apparatus for Estimation of Small Quantities by, 334
——, Rapid Electrolytic Analysis, Rotating, and Anode for, 334
Cattle and Poultry Food: "Artificial Preparation." Worcestershire County Council *v.* Notley Brothers (Law Report), 287
"Cellulose," Flour, Estimation of, in as a Measure of the Degree of Bolting, 309
Cement, Blast-Furnace Slag in Portland, Estimation of, 187
—— Plants, Dust Fall in the Neighbourhood of, Estimation of the, 372
Centrifugal Method, Urine, for Estimating Proteins in, 40
(Cereals, Coffee Mixtures, Estimation of Roasted, in), Analysis of Coffee, 85
Cerium, Pyrophoric Alloys of, and Iron, Analysis of, 415
Chambers, Electrically Regulated Constant Temperature, Thermo-Regulator for, 336
Cheese, Fat in, Butter, and Cream, Rapid Estimation of, 253
——, Water in, Estimation of, by Mai and Rheinberger's Method, 171
Chemical Analysis, Feeding-Stuffs, Valuation by Means of, of, 481
—— and Mineralogical, Granites and Gneisses, Quantitative, Composition of the, 98
Chemico-Physical Principles, Wine Analyses, Method of Expressing Results of, According to the Latest, 127
Chemistry, Electro-Titrimetric Method and its Application to General Analytical, 565
China Wood Oil, Examination of, 557
Chloral Hydrate, Flame Test for, 217
Chlorates, Acid Radicals in Chlorides, Perchlorates, Bromides, Bromates, and Fluorides, Application of Sodium Paratungstate and the Blowpipe Flame to the Estimation of the, 188
——, Nitrates, and Peroxides, Reduction Method of Analysing, 188
Chlorhydrocarbons, Chemical Analysis, The Properties of Some, and their Uses in, 4, 385
Chlorides, Acid Radicals in, Chlorates, Perchlorates, Bromides, Bromates, and Fluorides, Application of Sodium Paratungstate and the Blowpipe Flame to the Estimation of the, 188
——, Iron in Presence of, Permanganate Titration of, 415
Chlorine, Blood Serum, Estimation of, in, 223
——, Bromine Associated with, in Haloid Salts, Use of Telluric Acid in Estimation of, 331
——, Orthotolidine as Reagent for Colorimetric Estimation of Small Quantities of Free, 46
——, *o*-Tolidine and Starch Iodide Tests for Free, Effect of Ferric Salts and Nitrites on the, 454
——, Rain and Snow, and Nitrogen in, 190
——, Sulphuric Anhydride and of, The Chief Cause of the Loss of, by Incinerating Substances Containing These Constituents, 425
Cholesterol, Autenrieth and Funk, Estimation of, and Oxycholesterol by the Method of, 129
——, Fats, Estimation of, in, 310
Chromic Acid, Cyanogen Bromide, Action of, on, Detection of Bromides in Presence of Thiocyanates and Ferrocyanides, 412

Chromic Oxide, Chromium as, Estimation of, 97
Chromium, Chromic Oxide, Estimation of, as, 97
    Iron, Aluminium, ——, Zinc, and Manganese, Investigation of the Methods of Analysis and Separation of, 272
    Iron and Steel, Estimation of, and Manganese in, 454
    Oxidation in Alkaline Solution, Estimation of, by, 143
    Titanium and, Volumetric Estimation of, by Means of a Modified Reductor, 419
Chrysophanic Acid, Complex Medicinal Preparations, Some Reactions of, with Reference to its Detection in, 353
Cider, Tannin in, Estimation of, 127
—— Vinegar, Furfural in, 256
—— Vinegars Made by Generator Process, Composition of, 36
Cineol Content, Eucalyptus Oils, Notes on the Determination of the, of, 475
Citrate Method, Citric Acid Soluble Phosphoric Acid in Basic Slag, Estimation by the, and Lorenz's Method of, 377
Citric Acid, Cane Sugar in Beet Molasses, Determination of, by a Modification of the Double Polarisation (Clerget-Herzfeld) Method, Using, 394
————, Colour Reactions for, 395
————, Mineral Phosphates in, Solubility of, 146
———— Soluble Phosphoric Acid, Estimation in Basic Slag of, by the Citrate Method and Lorenz's Method, 377
    - Phosphoric Acid, Estimation in Basic Slag of, 377
Citrus Sinensis, Essential Oil of, 558
Clay, Humus in Heavy Soils, Determination of, 132
Clays, Rational Analysis of, 499
(Clerget-Herzfeld) Method, Cane Sugar, Muta-Rotation as a Source of Error in the Double Polarisation, of Determining, 258
——————————, Cane Sugar in Beet Molasses, Determination of, by a Modification of the Double Polarisation, Using Citric Acid, 394
Clinical, Dialysis Method in, and Biological Problems, Utility of the, 223
Clove, 1. Oils of Sassafras, Anise, Fennel, and Pimento, Hydrogen Value of Some Essential Oils and Essential Oil Products, 557
Coagulation, Rennet, Fat Content of Milk and Rate of, with, 357
Coal, Ash in, Relation of Composition of, to its Fusing Temperature, 370, 445
—— Gas, Calculated and Determined Calorific Values of, Difference between, 132
——, Solvents, Action on, of, 406
——, ———— for, 320
——, Sulphur Compounds of, and of Nitrogen in the Bomb Calorimeter, Oxidation of, and the Correction to Applied in Determining the Heating Value of, 563
——Tar Dyes in Wines, Detection of, 358
Cobalt, Colorimetric Estimation of, Nickel, Iron, and Copper, 326
———, Steel, Estimation of, in, 271
———, Steels, Estimation of, in High-Speed, 503
Cocaine, Reaction of, New, 481
"Cocoa Butter," "Cocoanut Oil" and, The Use of the Expressions, 30
Cocoa, Report on, and Cocoa Products, 123
"Cocoanut Oil," "Cocoa Butter," the Use of the Expressions, and, 30
Cocoanut Oil, Butter Fat, Detection of, in, by Means of Polenske's Distillation Method and Bömer's Phytosteryl Acetate Test, 254
Coffee, Analysis of (Estimation of Roasted Cereals in Coffee Mixtures), 85
———, Caffeine. Estimation in, of, 394
Cohune Nuts, Fat of, 396
Coinage, Zinc in Bronze, Estimation of, by Volatilisation, 236

Collargol, Organic Fluids, Estimation of Silver in, and, 278
Colloidal Silver, Silver, Estimation of, in, 279
Colloids, Effluents, Estimation of, in, and the Use of the Interferometer in Water Analysis, 325
Colophony, Fats and Soaps, Method of Estimating in, 181
———, Melting-Points of Constituents of, 256
Colorimeters, Two New, 282
Colorimetric Method, Nitrates and Nitrites, for Detecting and Estimating, 276
Colour, Methyl Alcohol, Reaction for the Detection of, 264
———, Reactions for Citric Acid, 395
———, Reactions of Organic Compounds with Trichloroacetic Acid, 410
——— Shades, Measurement and Nomenclature of, Industrial Applications of Various Processes and Systems for the, 149
Colouring Substances, Sesame Oil in Margarine containing, Detection of, 86
Combustion Furnace, Dennstedt, with Internal Electrical Heating, 150
——— Method, Rubber, for the Direct Estimation of, 368
———, Tellurium Dioxide for, Analyses, 192
Condensed Milk, Cane-Sugar and Lactose in, the Estimation of, by Means of Acid Mercuric Nitrate, 476
Conductivity, Beers, Electrical, of, 352
———, Milk, Electrical, of. Electrometric Determination of End-Point in Manufacture of Sweetened Condensed Milk, 174
——— Methods, Natural Waters, Applicability of, in Examination of, 149
Cone Oil, Cuban and Long-Leaf Pines, Leaf and Twig Oils of, and, of Long-Leaf Pine, 498
Coniferæ, Oils of the, Leaf and Twig, and Bark Oils of White Fir, 558
Constant Temperature Chambers, Thermo-Regulator for Electrically Regulated, 336
Cooking, Fats, the Changes in the Character of, during the Process of, 347
——— -Vessels, Nickel, 417
Copals, British West Africa, from, 396
Copper, Alloys, Analysis of, Tin, and Silicon, 500
——— ———, Approximate Melting-Points of Some Commercial, 98
———, Arsenic, and Iron, Iodimetry of, 116
———, Arsenic, Rapid Electrolytic Separation of, from, 371
———, Cobalt, Nickel, Iron, and, Colorimetric Estimation of, 326
——— Gauze, Zinc Determination, Silver-Plated, Electrode in, 382
——— -Iodide Method, Lactose, Estimation by the, of, 324
———, Oxydases and Minute Traces of, Guaiacum Resin as a Reagent for the Detection of, 39
———, Palladium, Separation of, from, and Iron, and Estimation of Palladium by Means of α-Nitroso-β-Naphthol, 55
——— Sulphate, Commercial, 414
———, Tin and, Groups, Separation of the, 371
Corn Silage, Methyl Alcohol in, Occurrence of, 549
Cottonseed, Husks in, Cakes and Meals, Estimation of, 173
Cottonseed Oil, Halphen's Test for, Modification of, 92
———, Ethyl Ester of Linolic Tetrabromide as a Product in the Analysis of, 532
Cream, Fat in, Cheese, Butter, and, Rapid Estimation of, 253
———, Gelatin in Sour, Detection of, 33
Creatine, Colorimetric Estimation of, 309
———, Folin Method for the Estimation of, and Creatinine, 41
Creatinine, Creatine and, Folin Method for the Estimation of, 41
(Creosote Oil), Wood Preservatives, Report of the Committee on, 497

Creosote, Timbers, Quantity and Character of, in Well-Preserved, 496
p-Cresol Tyrosinase Reaction, Peptides in Urine by the, Detection of, 555
Crucible, Ignitions, Double-Walled, for, 282
Cryolite, Differentiation of Natural from Artificial, 231
Crystallisation, Sulphates, Estimation of Water of, in, 193
Cupellation, Noble Metals, Loss of, on, 145
(Cupferron), Ammonium Salt of Nitrosophenylhydroxylamine, Use of, in Quantitative Separation of Titanium from Iron, 332
("Cupferron"), Nitrosophenylhydroxylamine, Separation of Zirconium from Iron and Aluminium with the Aid of the Ammonium Salt of, 565
Cupric-Reducing Power, Carbohydrates, Methods of Estimation of. III.: The, of the Pentoses, Xylose and Arabinose, 555
Cyanamide, Volumetric Estimation of, 365
Cyanides, Ferric Salts and, Volumetric Estimation of Ferricyanide in Presence of, 366
———, Ferro and Ferricyanides and Thiocyanates, Detection of, in Presence of, 365
Cyanogen Bromide, Chromic Acid on, Action of, Detection of Bromides in Presence of Thiocyanates and Ferrocyanides, 412
Cyanogenetic Plants of New South Wales, 442

"Dead" Rice, Composition of, 550
De-Aerated Liquids, Seal for Prevention of Aeration in, New, 509
Decomposition, Meats, Detection of, in, 130
Dehydration, Silica, and Separation of, 190
Densities, Aqueous Solutions of Certain, by a New and Precise Method, 61
Dennstedt Combustion Furnace, with Internal Electrical Heating, 150
Dextrose, Bone-Black, Absorption of, by, 497
———, Velocity of the Reduction Tests for, Influence of Metals Present in Urine on the, 131
Diastase, Pancreas, Assay of the Amylolytic, of, 318
Diastases, Plant Tissues, Quantitative Extraction of, from, 260
Diazonitro Reaction, Vegetable Oils, of, 228
Dichromate, Diphenylcarbohydrazide (Diphenylcarbazide), Application of, as Indicator in Titration of Iron with, 91
Digestive Ferments, Assay of, 359
Digitonin Method, Oils and Fats and Lanolin, Estimation of the Unsaponifiable Constituents of, by Means of the, 310
Dimethyl Phosphates, Rare Earths, of the, (Fractionation of Rare Earths), 146
Dimethylglyoxime, Nickel, Estimation of, by Means of, 375
Diphenylamine, Reagent for Oxidising Substances, Use of Diphenylbenzidine in Place of, as a, 320
Diphenylbenzidine, Oxidising Substances, Use of, in Place of Diphenylamine as a Reagent for, 320
Diphenylcarbohydrazine (Diphenylcarbazide), as Indicator in Titration of Iron with Dichromate, Application of, 91
Direct Combustion Method, Steel, Determination by the, of Carbon in, 560
Disinfectants, Method for Determining the Value of, 261
Dissolved Oxygen, Water, Estimation of, in, 55
———, ———, Field Method for Determining, in, 234
Distillation, Fractional, Still for Exact Measurement of Boiling-Point during, 456
——— Method, Polenske's and Bömer's Phytosteryl Acetate Test, Detection of Coconut Oil in Butter Fat by Means of, 254
Distribution Coefficients, Extraction by Means of Immiscible Solvents, Study of

Methods of, from Point of View of: 1. (Analysis of Vanilla Extracts and Extraction of Acetanilide, Saccharin, and Caffeine, 354
Dorton Spa, The Water of, 210
Double Polarisation (Clerget-Herzfeld) Method, Cane Sugar in Beet Molasses, Determination of, by a Modification of the, Using Citric Acid, 394
——————————, Cane Sugar, Muta-Rotation as a Source of Error in the, of Determining, 258
Double Polarisation Process, Cane-Sugar in Beet Products, Comparative Study of Different Methods of Determining, by the, 435
Double-Walled Crucible, Ignitions, for, 282
Drugs, Carbon Tetrachloride, the Extraction and Estimation of the Active Principles of, and Medicinal Plants, Use of, 255
———, Synthetic, New Remedies, Photographic Developers, etc., Index List of Trade Names of, 533
———, Volatile Oils in Aromatic Plants, and Spices, Estimation of, by the Tailameter, 35
Dry Strengths, Paper and Paper Yarns, Wet and, of, 322
Drying Oils, Insoluble Bromide Test for, Notes on the, 28
Drying-Oven, Temperatures, Non-Uniformity of, 457
Drying, Test Purposes, Apparatus for the, of Paper for, 508
Duck, Albumins, and Hen Egg, Differentiation between, for Commercial Purposes, 222
Dust, and Bacteria Samples of Air, Apparatus for Taking, 283
——, Cement Plants, Estimation of the, Fall in the Neighbourhood of, 372
Dyes, Wines, Detection of Coal-Tar, in, 358
[Dyestuffs], Report of the, "Fastness Committee," 183
Dynamical Methods, Vapour Pressures, New Method for the Determination of, and an Examination of a Source of Error in Certain, 511

Earth-Nut Butter, 481
Earths, Hydroxides, Errors in the Estimation of Rare, as, 330
———, Rare, Dimethyl Phosphates of the (Fractionation of Rare), 146
———, Yttrium, Separation of Yttrium from the, Part I., 280; Part II., 420
East Africa Protectorate, Tobaccos from the, 125
East India, Wet Salted, Goat Skins, Analysis of the Saline Matter Adhering to the Flesh Side of, 402
Effluents, Colloids, Estimation in, of, and the Use of the Interferometer in Water Analysis, 325
Egg, Hen and Duck, Albumins, Differentiation between, for Commercial Purposes, 222
Eggs, Boric Acid in Milk and in, Occurrence of, 123
Electrical Conductivity, Beers, of, 352
————, Internal, Heating, Dennstedt Combustion Furnace with, 150
————, Milk, Conductivity of, Electrometric Determination of End-Point in Manufacture of Sweetened Condensed Milk, 174
————, Thermoregulator, Contact Vapour-Pressure, 154
Electro-Analysis, White Metals, New Method for, of, 52
————Analytical Studies, 97
————Titrimetric Method, Analytical Chemistry, and its Application to General, 565
Electrode, Zinc Determination, Silver-Plated Copper Gauze, in, 382
Electrolysis, Small Quantities, Apparatus for Estimation by, with the Rotating Cathode of, 334
Electrolytic Analysis, Rotating Cathode and Anode for Rapid, 334
———— Thermostats, Method for Heating and Regulation of, 64

End-Point, Titrations with Fehling's Solution, New Indicator for Use in Determining, in, 133
Enzyme Action, Castor Bean Urease, Experiments with, (Studies on. XI.), 553
Enzyme Work, "Tricresol" as a Substitute for Toluene in, 263
Enzymes, Acidity in Meal, Grist, and Bread, Estimation of, Taking into Account the Action of Bacteria and, 38
————, Alfalfa (Lucerne), Present in, 552
Errata, 1914, 121 and 516
Error, "Thomas" Meal, Sources of, in the Analysis of, 377
Errors, Gas Analysis, in, Due to Assuming that the Molecular Volumes of All Gases are Alike, 231
Essential Oil, *Citrus Sinensis*, of. 558
————, *Ocymum Pilosum*, Roxb., of, 438
———— Products, Essential Oils and, Hydrogen Value of Some. 1. Oils of Sassafras, Anise, Fennel, Clove, and Pimento, 557
Essential Oils, Analysis of, 396
————, Esters, Estimation in, of, 436
————, Hydrogen Value of Some, and Essential Oil Products. 1. Oils of Sassafras, Anise, Fennel, Clove, and Pimento, 557
————, Various Countries, from, 397
Esters, Essential Oils, Estimation of, in, 436
———, Phosphoric Acid, Estimation of Phosphoric Acid in the Presence of, 559
Ether, Liquids, Extraction of, Apparatus for, 239
Ethereal Sulphates, Urine, Volumetric Method for the Estimation of Inorganic and, in, 319
Ethyl Alcohol, Methyl Alcohol and, Detection of, 184
————, Specific Reaction of, 319
——— Ester, Cottonseed Oil, of Linolic Tetrabromide as a Product in the Analysis of, 532
Eucalypts, Tasmanian and Australian, Correlation between the Specific Characters of the, 435
Eucalyptus Oils, Cineol Content of, Notes on the Determination of the, 475
Explosives, Heat Test of, 184
————, Permissible, Tests of, *Report*, 286
Extract, Malt, Determination of, 407
-, 356
-, Wine, Estimating, in, 221
Extracted Oils, Carbon Disulphide, Detection in, of, 180
———— Paprika, Detection of, 174
Extraction Apparatus, 61
————, New, 240
-, Simple, 381
————, Immiscible Solvents, Study of Methods of, from Point of View of Distribution Coefficients, by Means of. 1. (Analysis of Vanilla Extracts and, of Acetanilide, Saccharin, and Caffeine), 354
————, Solvents of High Specific Gravity, Apparatus Specially Designed for, 240
Extractor, Fat, A Simple, 295
Extracts, Nitrogen in Vegetable, Yeast, and Meat, Distribution of, 444
————, Phosphates, Estimation in Soil, of, 329
————, Soils and Soil, Estimation of Carbon in, 449

Fachini-Dorta Method, Oils and Fats, The, of [Approximately] Separating Liquid from Solid Fatty Acids in, 389
"Fastness Committee," Report of the [Dyestuffs], 183

Fat, Cheese, Butter, and Cream, Rapid Estimation of, in, 253
—, Cohune Nuts, of, 396
— Extractor, A Simple, 295
—, Milk, Content of, and Rate of Coagulation with Rennet, 357
————, Nephelometric Method for the Estimation of, in, 356
—, Stag's, Characteristics of, 32
Fats, Acetyl Value of Oils, etc., Estimation of the, 362
——, Animal and Vegetable, Unsaponifiable Constituents of Mowrah Fat and the Detection of this Fat in, 310
——, Animal, in Vegetable, Detection of, 32
——, Cholesterol, Estimation in, of, 310
——, Colophony in, and Soaps, Method of Estimating, 181
——, Cooking, The Changes in the Character of, during the Process of, 347
——, Digitonin Method, Estimation of the Unsaponifiable Constituents of Oils and, and of Lanolin by Means of the, 310
——, Hydroxy Fatty Acids in, Estimation of, by Means of Magnesium Methyl Iodide, 41
——, Iodine Value of, Comparison of Various Methods for Determining the, 406
————————, Estimation of the, and Action of Halogens on Oleic Acid, 311
——, Nickel in, Detection of, 254
——, Oils and, The Fachini-Dorta Method of [Approximately] Separating Liquid from Solid Fatty Acids in, 389
——, Phytosterol in Animal, Detection of, 84
——, Vegetable, in Animal, Detection of, 32
Fatty Acids, Mixtures of, Melting and Solidifying Points of, and Use of these Points to Determine Composition of Such Mixtures, 448
————, Note on the, and Detection of Arachidic Acid, 122
————, Oils and Fats, The Fachini-Dorta Method of [Approximately] Separating Liquid from Solid, in, 389
————, Soaps, Estimation of, in, 311
Feeding-Stuffs, Chemical Analysis, Valuation of, by Means of, 481
—————, Hydrocyanic Acid in, The Estimation of, and its Occurrence in Millet and Guinea Corn, 430
—————, Water in Foods and, Rapid Estimation of, 36
Fennel, Hydrogen Value of Some Essential Oils and Essential Oil Products. I. Oils of Sassafras, Anise, Clove, and Pimento, 557
Ferment Reactions, Goat's Milk, Schardinger's Formaldehyde-Methylene Blue Reaction, and Other, of, 218
(Fermentation-Reductase), Reductase, Test, 224
————— Test at 46° C. as an Aid in Water Examination, 129
Ferments, Digestive, Assay of, 359
Ferric Ammonium Sulphate, Casein, Estimation by Means of, of, 496
——— Chloride, Titanium, Volumetric Estimation of, by Means of, 563
——— Salts, Ferricyanide in Presence of, and Cyanides, Volumetric Estimation of, 366
—————, *o*-Tolidine and Starch Iodide Tests for Free Chlorine, Effect of, and Nitrites on the, 454
—————, Titrating, New Method of, 189
Ferricyanide, Ferric Salts and Cyanides, Volumetric Estimation of, in Presence of, 366
Ferricyanides, Cyanides in Presence of Ferro- and, and Thiocyanates, Detection of, 365
Ferrocyanide, Alkali Bromate, Estimation of, by Titration with, 366

Ferrocyanides, Bromides in Presence of Thiocyanates and, Detection of, Action of Chromic Acid on Cyanogen Bromide, 412
Detection of, 187
Cyanides in Presence of, and Ferricyanides and Thiocyanates, 365
Fibre, Crude, Method for the Estimation of, 367
Fibre, Finely Powdered Substances, Estimation of Crude, in, 218
Field Method, Dissolved Oxygen in Water, for Determining, 234
Filament, Concentrated, Tungsten Lamp, Uses in the Laboratory of the, 194
Filter from which a Precipitate may be Removed Quantitatively, 151
Filtration, Suction, Device to Assist, by, 508
Finely Powdered Substances, Extraction of, 420
Finishing, Potato Starch, Rapid Testing of, for, Purposes, 94
Fir, White, Oils of the *Coniferæ* Leaf and Twig, and Bark Oils of, 558
Fire-damp Whistle, 193
Fishes, Soft Roe and Hard Roe of, Composition of the, 261
Flame Test, Chloral Hydrate, for, 217
Flax, Waxes from, and Hemp, 560
Flour, "Cellulose" in, Estimation of, as a Measure of the Degree of Bolting, 309
——, Gliadin in, and Gluten, Quantitative Estimation of, 33
——, Notes on, 1. Acidity of; 2. Natural and Artificial Bleaching of; 3. Sulphates and Lime in, 519
——, Proteins in Wheat, Estimation of the, 550
——, Rye, Detection in Wheat, of Small Quantities of, 400
——, Sulphates, Note on the Presence in, of, 429
——, Wheat, Detection of Small Quantities of Rye Flour in, 400
————, Milling of, and Testing of, 358
————, Quantitative Estimation of the Proteins Soluble in Salt Solutions in, 262
Flours, Ivory-Nut Meal in, and Fodders, Estimation of, 218
Fluorescence, Petroleum Distillates, of, 446
Fluorides, Acid Radicals in Chlorides, Chlorates, Perchlorates, Bromides, Bromates, and, Application of Sodium Paratungstate and the Blowpipe Flame to the Estimation of, 188
————, Aluminium, Precipitation of, in Presence of, 269
————, Soluble, The Action of Weak Acids on, 248
Fluorine, Mineral Waters, in, 401
————, Reagents, Occurrence of, in, 231
Fodders, Ivory-Nut Meal in Flours and, Estimation of, 218
Folin Method, Creatine and Creatinine, for the Estimation of, 41
Fontactoscope, Spring Waters, for Measuring the Radio-Activity of, 423
Food, Cattle and Poultry: "Artificial Preparation." Worcestershire County Council *v.* Notley Brothers (Law Report), 287
—— Materials, Phosphates in the Ash of, Volumetric Estimation of, 234
FOODS AND DRUGS ANALYSIS, ABSTRACTS, 31, 84, 122, 171, 215, 252, 308, 352, 393, 433, 480, 532
Foods, Boric Acid in, Estimation of, 123
——, Inspectors of, Report on the Work of, for the Year 1912-1913, 195
——, Milk Production, Method for Calculating the Value of, from the Point of View of, 171
——, Saccharin, Estimation in, of, 86
——, Water in, and Feeding Stuffs, Rapid Estimation of, 36
Formaldehyde - Methylene Blue Reaction, Schardinger's, and Other Ferment Reactions of Goat's Milk, 218

Formaldehyde, Phenol in the Presence of Hexamethylenetetramine and, Determination of, 266
————, Schiff's Reagent Containing Hydrochloric Acid, Detection of, and of, Compounds by Means of, 172
————, Titration with Acid after Treatment with, Estimation of Amino-Acid and Polypeptide-Nitrogen in Barley, Malt, and Beer, by, 257
Formic Acid, Method of Estimating, 320
————, Nitrates, Reaction with, of, 233
————, "Vacuum Distillation" Method for the Detection of, 321
Fractional Distillation, Gas Analysis by, at Low Temperatures, 414
———, Pine Woods, of Mixed, 48
Liquefaction, Rice Starch, of, 440
Freezing-Point, Thermometry, of Benzene as a Fixed Point in, 510
Fresenius's Method, Nitrites, On, for Determining Small Quantities of, and its Sensitiveness Compared with the *m*-Phenylene-Diamine Reaction, 350
Fuels, Water in Solid and Liquid, Estimation of, by Distillation with Xylene, 141
Fulminate, Mercury, Electrolytic Estimation of, in, 275
Fume and Smoke Monitors, Instantaneous Thermostat and, Precipitators, and Recorders, 567
Funk and Autenrieth, Cholesterol and Oxycholesterol, Estimation by the Method of, of, 129
Furfural, Cider Vinegar, in, 256
Furnace, Dennstedt Combustion, with Internal Electrical Heating, 150
Fusing Temperature, Coal, Relation of Composition of Ash in, to its, 370

Galena, Sulphuric Acid, Velocity of Solution of Blende and, in Dilute, 452
Gallium, Aluminium, Presence of, in Commercial, 53
Gas, Ammonia, Estimation in Illuminating, of, 369
—— Analysis Apparatus, 421
————, Errors in, Due to Assuming that the Molecular Volumes of All Gases are Alike, 231
————, Fractional Distillation at Low Temperatures, by, 414
————, Manostat for Use in, 458
—— Generator, Permanent Use, for, 239
—— Mixtures, Complex, Apparatus for Analysis of, 566
——Sampling, Automatic, Apparatus, with Some Observations on Sampling, 103
——Velocities, Pitot-Tubes for Measurement of, 456
——, Very Small Quantities of, Analysis of, Application to Air Analysis, 413
——Volumetric Estimation, Urine, of Free and Combined Amino-Nitrogen in, 175
Gaseous Hydrocarbons, Mineral Springs, Detection and Estimation of, in the Water of, 227
Gases, Bromine in, and Vapours, Detection of. VII. Communication, 271
——, Mixed, Improved Apparatus for the Analysis of, 391
——, Zeiss Interferometer for the Analysis of, 103
Gauge, Pressure, New Differential, 60
Gelatin, Sour Cream, Detection of, in, 33
———, Sulphur Dioxide in, 87
Gelatins, Paper-Making, The Examination of Commercial, with Regard to their Suitability for, 20
Generator, Nitrogen, 422
———— Process, Cider Vinegars Made by, Composition of, 36
Geographical Sources, Hops, Aroma of, Study of the Volatile Oil with Relation to the, of the Hops, 355
German Standards, Malt Analysis, New, for, 436

Gingerbread, Sugars in, and Similar Products, Separation and Estimation of, 358
Gliadin, Flour and Gluten, Quantitative Estimation of, in, 33
Glue, Sizings, Identification of Rosin and, 411
Gluten, Gliadin in Flour and, Quantitative Estimation of, 33
Glycerines, Glycerol in Technical, and Soap Lyes, Estimation of, 181
Glycerol, International Standard Methods, 1911, Analysis of Crude, by the. Estimation of Organic Residue, 34
———, Technical Glycerines and Soap Lyes, Estimation of, in, 181
———, Wine, Estimation of, in, 87
Glycogen, Yeast, Determination of, in, 129
Gneisses, Granites and, Quantitative Mineralogical and Chemical Composition of the, 98
Goat Skins, East India, Analysis of the Saline Matter Adhering to the Flesh Side of Wet Salted, 402
Goat's Milk, Schardinger's Formaldehyde-Methylene Blue Reaction and Other Ferment Reactions of, 218
Golden Sulphide, Antimony, Analysis of, of, 186
Government Laboratory, Work of the, for the Year Ended March 31, 1914, Report of the Government Chemist upon the, with Appendices, 458
Granites, Gneisses and, Quantitative Mineralogical and Chemical Composition of the, 98
Graphite, Sulphuretted Hydrogen from Artificial, 67
Grist, Acidity in Meal, and Bread, Estimation of, Taking into Account the Action of Bacteria and Enzymes, 38
Gru-Gru Oil, 124
Guaiacol, Urine, Detection of, in, 360
Guaiacum, Oxydases and Minute Traces of Copper, Resin as a Reagent for the Detection of, 39
Guinea Corn, Millet and, The Estimation of Hydrocyanic Acid in Feeding-Stuffs, and its Occurrence in, 430
Guncotton, Nitrometer, Determination of Nitrogen in, by Means of the, 409

Hæmatin, Blood, Estimation of, in, 262
Halides, Hydroxyl Groups, Pyridine as a Solvent in the Estimation of, by Means of Alkyl Magnesium, 186
Halogen Oxyacids, Semicarbazide, Estimation of, by its Interaction with Halogens and, 323
Halogens, Oleic Acid, Action of, on and Estimation of the Iodine Value of Fats, 311
———, Organic Compounds, Estimation of, in, 367
———, Carbon, Estimation of, by the Wet Way. Simultaneous Estimation of, 132
———, Semicarbazide, Estimation of, by its Interaction with, and Halogen Oxyacids, 323
Haloid Salts, Bromine Associated with Chlorine in, Use of Telluric Acid in Estimation of, 331
Halphen's Test, Cotton-Seed Oil, Modification of, for, 92
Hardness, Butter Fat, Apparatus for Determining the, of, 282
———, Water, Estimation of, in, 379
Heat, California Petroleums, Specific, of, 499
—— Test, Abel, Some Observations on the, 44
———, Explosives, of, 184
Heats, Liquids, New Method for Determining the Specific, of, 510
Heating, Value of Coal, Oxidation of Sulphur Compounds of Coal, and of Nitrogen in the Bomb Calorimeter, and the Correction to be Applied in Determining the, 563
Hemp, Flax and, Waxes from, 560

Hen, Albumins, Differentiation between, and Duck Egg, for Commercial Purposes, 222
Herzig and Meyer's Reaction, Proteins and Amino-Acids, Applied to, 317
Hevea Rubber, 30 per cent., Insulating Compound, Analytical Procedure for, 137
Hexamethylenetetramine, Phenol in the Presence of, and Formaldehyde, Determination of, 266
————————, Wine or Milk, Detection of, in, 125
High Dilutions, Ozone, Nitrogen Peroxide, and Hydrogen Peroxide at, Estimation and Distinction of, 189
—— Temperature Thermostat, 153
Higher Oxides, Lead, Analysis of, of, 328
Histidine, Colorimetric Estimation of, 92
Honey, Sulphate in, and Other Substances Rich in Sugar, Estimation of, 259
Hop Bitter Substances, Carbon Tetrachloride, Estimation of, by Cold Extraction with, 125
Hops, Aroma of, Study of the Volatile Oil with Relation to the Geographical Sources Sources of the, 355
——, Nitrogenous Constituents of, 492
Humus, Soils, Determination of, in Heavy Clay, 132
Husks, Cottonseed Cakes and Meals, Estimation of, in, 173
Hydrocarbon Content, Concentrated Lemon Oils, Determination of the, of, 185
Hydrocarbons, Sulphur Dioxide, Separation of, by Means of Liquid, 321
————, Water of Mineral Springs, Detection and Estimation of Gaseous, in the, 227
Hydrochloric Acid, Alkali Carbonates, Convenient Standard End-Point in Titration of, with, 268
——————, Formaldehyde and Formaldehyde Compounds, Detection of, by Means of Schiff's Reagent Containing, 172
Arsenic in, and Sulphuric Acids, Estimation of, 370
Hydrocyanic Acid, Alkali Cyanides, New Method for Determination of, and, 448
——————, Feeding-Stuffs, The Estimation of, in, and its Occurrence in Millet and Guinea Corn, 430
——————, Linseed, The Rate of Liberation of, from, 70
Hydrogen, Catalytic Reductions with, Apparatus for, 333
———— Peroxide, Ozone, Nitrogen Peroxide, and, at High Dilutions, Estimation and Distinction of, 189
————, "Silicol" and Other Silicon Compounds Used for the Production of, Analysis of, 147
— Sulphide, Small Quantities of, Colorimetric Estimation of, 325
, Water, New Apparatus for Estimation of, in. Part I., 457
————, Sulphuretted, from Artificial Graphite, 67
———— Value, Essential Oils and Essential Oil Products, of Some. I. Oils of Sassafras, Anise, Fennel, Clove, and Pimento, 557
Hydrolysis, Lævulosans, The, of, and its Application in Plant Analysis, II. and III., 88
Hydroxides, Rare Earths as, Errors in the Estimation of, 330
Hydroxy Fatty Acids, Fats, Estimation of, in, by Means of Magnesium Methyl Iodide, 41
Hydroxybutyric Acid, Estimation of, 184
Hydroxyl Groups, Pyridine as a Solvent in the Estimation of, by Means of Alkyl Magnesium Halides, 186
Hypochlorite Disinfection, Water-Supplies, Results of the, of. A Comparison of the Reduction of the Different Members of the *Bacillus Coli* Group, 443
Hypophosphorous Acid, Arsenic, Quantitative Separation of, from the Metals by Means of, 269

Ignitions, Double-Walled Crucible for, 282
Illuminating Gas, Estimation of Ammonia in, 369
Immiscible Solvents, Methods of Extraction by Means of, Study of, from Point of View of Distribution Coefficients. I. (Analysis of Vanilla Extracts and Extraction of Acetanilide, Saccharin, and Caffeine), 354
Index List, Synthetic Drugs, New Remedies, Photographic Developers, Etc., of Trade Names of, 533
India, Corrected Sikes's Tables in Use in, for Determining the Proof Strengths of Spirits at Temperatures from 40° F. to 100° F. by Sikes's Hydrometer, Method by which were Calculated the, 532
India-Rubber, Sulphur in, Estimation of Total, 368
Indicator, Dry Starch, Soluble in Cold Water, for Use as an, Preparation of, 451
————, End-Point in Titrations with Fehling's Solution, New, for Use in Determining, 133
————, Methyl Red as an, 518
Indicators, Analytical Chemistry, Radio-Elements as, in, 57
————, Natural, Some, 499
————, Phthaleïns, Properiies of the, as, 136
Indigo-Forming Substances, Urine, in, 39
Industrial Applications, Colour Shades, of Various Processes and Systems for the Measurement and Nomenclature of, 149
Infant Feeding, Local Government Board, Reports to the : I. On the Use of Proprietary Foods for; II. On the Analysis and Composition of Some Proprietary Foods for Infants, 464
Inks, Printing, and their Analysis, 450
INORGANIC ANALYSIS, ABSTRACTS: 49, 96, 142, 186, 230, 268, 324, 369, 412, 452, 499, 560
———— Sulphates, Urine, Volumetric Method for the Estimation of Ethereal and, in, 319
Insoluble Bromide Test, Drying Oils, Notes on the, for, 28
———————— Value, Oils, Note on the, of, 388
————————, ——, The, of, and its Determination, 297
INSTITUTE OF CHEMISTRY; 158, 197, 242, 384
Interferometer, Gases, Zeiss, for the Analysis of, 103
————, Water Analysis, Estimation of Colloids in Effluents and the Use of the, in, 325
INTERNATIONAL ATOMIC WEIGHTS, 1914, 3
Invertase Solutions, Top and Bottom Yeasts, Method of Preparing Strong, from, 443
Iodate, Periodate in Presence of, and Iodide, Estimation of, 56
Iodide, Periodate in Presence of Iodate and, Estimation of, 56
Iodimetric Estimation, Iodide Iodine, of, 99
Iodimetry, Arsenic, Copper, and Iron, of, 116
Iodine, Organic Substances, Estimation of, in, 367
Iodine Value, Fats, Comparison of Various Methods for Determining the, of, 406
Insulating Compound, 30 per cent. Hevea Rubber, Analytical Procedure for, 137
Iodide Iodine, Iodimetric Estimation of, 99
Iodine Value, Oleic Acid, Action of Halogens on, and Estimation of the, of Fats, 311
Iridium, Platinum-Iridium Alloys, The Determination of, in, 340
Iron, Aluminium, Chromium, Zinc, and Manganese, Investigation of the Methods of Analysis and Separation of, 272
——, Carbon in Steel and, Determination of, by the Barium Carbonate Titration Method, 373
——, Cerium and, Analysis of Pyrophoric Alloys of, 415
—— Chlorides, Permanganate Titration of, in Presence of, 415
—— Chromium and Manganese, Estimation in, and Steel of, 454

Iron, Cobalt, Nickel, and Copper, Colorimetric Estimation of, 326
—— Diphenylcarbohydrazide (Diphenylcarbazide), Application of, as Indicator in Titration of, with Dichromate, 91
—— Manganese, Separation of, and, 272
—— Ores, Estimation in, of, 144
—— Palladium by Means of α-Nitroso-β-Naphthol, Estimation of, and Separation of Palladium from Copper and, 55
—— Permanganate Titration, Electrolytic Reduction of, for, 328
—— Phosphoric Acid, Determination of, in Presence of, 416
——, Quantitative Separation of Titanium from, Use of Ammonium Salt of Nitrosophenylhydroxylamine ("Cupferron") in, 332
—— Sediment in Bottled Mineral Waters, Formation of. Estimation of Silica, and Aluminium in Mineral Waters, 126
——, Water, Colorimetric Estimation of, in, 333
——, Zirconium from, and Aluminium, Separation from, with the Aid of the Ammonium Salt of Nitrosophenylhydroxylamine ("Cupferron"), 565
Isoprene, Physical Constants of, 406
————, Synthetic Caoutchouc from, Note on, 179
Italian Tobacco, Barium and Arsenic in, 43
Ivory-Nut Meal, Flours and Fodders, Estimation of, in, 218

Jams, Salicylic Acid, Estimation in, of, 219
Juice, *Carica Papaya*, Standardisation of Dried, 170

Kjeldahl's Method, Nitrogen, Modifications of the, of Estimating, 227
————————, Nitro-Compounds, Estimation of Nitrogen in, by, 93
Kola Preparations, Caffeine, Estimation in, of, 31

Laboratory Method, Liquid Mixtures, Rapid, of Measuring Partial Vapour Pressures of, 569
Lacmoid, "Lacmosol," the Active Constituent of, 556
"Lacmosol," Lacmoid, the Active Constituent of, 556
Lactic Acid, Blood and Other Physiological Liquids, Estimation of Carbohydrates and, in, 551
—————, Liquids Containing Protein, Estimation of, in, 133
—————, Wine, Estimation by Moslinger's Barium Chloride Method of, in, 397
—————, ————, Estimation of, in, 37
Lactose, Ammonium Sulphate, Influence of, on the Rotation of, 133
————, Cane Sugar in the Presence of, Estimation of, 363
————, Condensed Milk, The Estimation by Means of Acid Mercuric Nitrate of Cane Sugar and, in, 476
————, Copper-Iodide Method, Estimation of, by the, 324
————, Milk, Polarimetric Estimation of, in, 399
Lævulosans, Plant Analysis, The Hydrolysis of, and its Application in: II. and III., 88
Lævulose, Sugars, Detection and Estimation of, in Presence of Other, 323
Lamp, Concentrated Filament Tungsten, Uses in the Laboratory of the, 194
Landsberger-Sakurai Method, Molecular Weight, Improved Apparatus for the Determination of, by the, 152
Lanolin, Unsaponifiable Constituents of Oils and Fats and of, Estimation by Means of the Digitonin Method of the, 310
Lard, Beef and Mutton Fats in, Detection by Bömer's Method of, 253
——, Beef Fat in, Detection by Polenske's and Bömer's Methods of, 216
——————, Detection in, of, 84, 171

Lathering, Soap, Capacity of, 312
Lead Carbonate, Alkali Carbonate Solutions, Behaviour of, Basic Lead Carbonate, and Lead Sulphate towards, 53
——, Higher Oxides of, Analysis of, 328
——, New and Sensitive Reaction of, 274
—— Peroxide, Lux's Method for the Estimation of, Note on, 328
——, Reagents for, Sensitiveness of Various, 374
—— Red, Analysis and Composition of, 375
—— Salts, Sodium Hydrogen Carbonate Solutions, Action of, on Difficultly Soluble, 143
——, Small Quantities of, Estimation of, 274
—— Sulphate, Alkali Carbonate Solutions, Behaviour of Lead Carbonate, Basic Lead Carbonate, and, towards, 53
——, Tungsten, Effect on Ammonium Molybdate Assay for, of, 192
——, Urine in Cases of, Poisoning, Estimation of Small Quantities of, particularly in, 495
——, Volumetric Estimation of, 373
Leaf and Twig Oils, *Coniferæ*, Oils of the, and Bark Oil of White Fir, 558
————————, Cuban and Long-Leaf Pines, of, and Cone Oil of Long-Leaf Pine, 498
Leaves, Senna, Constituents of, 35
Lemon Oils, Hydrocarbon Content of Concentrated, Determination of the, 185
Lime, Soil, Determination of, Requirements of, 450
——, Soils, Estimation of the, Requirement of, 90
Linolic Tetrabromide, Cottonseed Oil, Ethyl Ester of, as a Product in the Analysis of, 532
Linseed, Hydrocyanic Acid, The Rate of Liberation from, of, 70
——— Oil, Oxygen Absorption of, during Drying, Gravimetric Estimation of the, 92
Lipoids, Blood Serum, Estimation of, in, 176
Liquid Mixtures, Partial Vapour Pressures of, Rapid Laboratory Method of Measuring, 569
Liquids, Carbohydrates and Lactic Acid in Blood and Other Physiological, Estimation of, 551
———, De-Aerated, New Seal for Prevention of Aeration in, 509
———, Ether Extraction of, Apparatus for, 239
———, Lactic Acid, Estimation of, in, Containing Protein, 133
-, Specific Heats of, New Method for Determining the, 510
---, Very Volatile, Apparatus for the Combustion of, 566
--, Zelmanowitz's Apparatus, Modification of, for Extracting, 64
Liquorice Powder, Compound, The Composition and Analysis of, 160
List, Index, of Trade Names of Synthetic Drugs, New Remedies, Photographic Developers, etc., 533
Loading, Caoutchouc Mixtures, Estimation of, in, according to the Standards of the Verband Deutscher Elektrotechniker, 91
Local Government Board, Reports to the: I. On the Use of Proprietary Foods for Infant Feeding. II. On the Analysis and Composition of Some Proprietary Foods for Infants, 464
Long-Leaf Pine, Leaf and Twig Oils of Cuban and Long-Leaf Pines and Cone Oil of, 498
Lorenz's Method, Citric Acid Soluble Phosphoric Acid in Basic Slag, Estimation of, by the Citrate Method and, 377
Lothar Meyer's Apparatus, Mercury, Modified, for the Purification of, 151

Lubricants, Tests for, Standard, 557
(Lucerne), Alfalfa, Enzymes Present in, 552
Lungs, Silicotic, Ash of, 359
Lux's Method, Lead Peroxide, Note on, for the Estimation of, 328

Magnesium, Calcium and, in the Same Solution, Titration of, 51
————, ———, Separation from, of, 142
———— Methyl Iodide, Hydroxy Fatty Acids in Fats, Estimation of, by Means of, 41
———— Sulphate, Detection of, in, 219
Mai and Rheinberger's Method, Cheese, Estimation of Water in, by, 171
Malic and Succinic Acids, Colour Reaction of, 186
Mallory Connective Tissue Stain, Ruthenium Red and the, Treatment of Sinews, particularly by the Application of, 89
Malt, Amino-Acid and Polypeptide-Nitrogen in Barley, and Beer, Estimation of, by Titration with Acid after Treatment with Formaldehyde, 257
—— Analysis, New German Standards for, 436
—— Extract, 356
——————, Determination of, 407
—— Wine, 128
——, Worts, and Beers, Temperature Corrections to be Applied to the Specific Gravities of, 256
Manganese Carbonate, Strychnine, Identification by Means of, of Traces of, 357
————, Iron, Aluminium, Chromium, Zinc, and, Investigation of the Methods of Analysis and Separation of, 272
——, Iron and, Separation of, 272
——, ———— Steel, Estimation of Chromium and, in, 454
——, Marshall's, Reaction, 274
—— Sulphide and the Estimation of, 417
——— Manganese, 417
——, Water, Colorimetric Estimation of Small Quantities of, in, 54
—————— Traces of, in, 380
Mannitol, Estimation of, 93
————, ——————, Methods for the, 407
Manometric Method, Urea, for the Estimation of, 553
Manostat, Gas Analysis, for Use in, 458
Maple Products, Analysis of, 11, 85
Margarine, Butter Fat Content of, 216
————, Sesame Oil, Detection of, in, Containing Colouring Substances, 86
Marshall's Manganese Reaction, 274
Meal, Acidity in, Grist, and Bread, Estimation of, Taking into Account the Action of Bacteria and Enzymes, 38
——, Flours and Fodders, Estimation of Ivory-Nut, in, 218
——, "Thomas," Sources of Error in the Analysis of, 377
Meals, Cottonseed Cakes and, Estimation of Husks in, 173
Meat, Vegetable, Yeast, and, Extracts, Distribution of Nitrogen in, 444
Meats, Decomposition, Detection in, of, 130
Medicinal Plants, Carbon Tetrachloride in the Extraction and Estimation of the Active Principles of, and Drugs, Use of, 255
———— Preparations, Chrysophanic Acid, Some Reactions of, with Reference to its Detection in Complex, 353
Melting Point, Tars, Relation between the, and Viscosity of Refined, 369
———— -Points, Colophony, of Constituents of, 256

Melting-Points, Mixtures of Fatty Acids, Solidifying and, of, and Use of these Points to Determine Composition of Such Mixtures, 448
————, Silicates, Determination of, of, 236
————, Thermometer Bulb Method, Determination of (of Fats, etc.), by the, 61
Mercuric Chloride, Caffeine, Theobromine, Theophylline, and their Derivatives, Micro-Chemical Reaction with, of, 217
Mercuric Chloride, Volumetric Estimation of, 233
———— Iodide, Tablets, Estimation of, in, 493
———— Oxide, Carbon Monoxide, Estimation of, by Means of Yellow, 226
Mercury, Fulminate, Electrolytic Estimation of, in, 275
————, Lothar Meyer's Apparatus for the Purification of, Modified, 151
————, Method of Estimating, New, 144
————, Urine, Electrolytic Method for Estimation of, in, 225
Metabolism, Milk, Studies in Bacterial, in, 494
Metal, Monel, Analysis of a Sample of, 561
Metallic Tungsten, High-Grade, Analysis of, 505
Metals, Cupellation, Loss of Noble, on, 145
————, Dextrose, Influence of, Present in Urine on the Velocity of the Reduction Tests for, 131
————, Electro-Analysis of White, New Method for, 52
————, Hypophosphorous Acid, Quantitative Separation of Arsenic from the, by Means of, 269
Methoxyl Estimations, Note on, 134
————, Sulphur, Estimation of, in Compounds Containing, 265
Methyl Alcohol, Boric Acid, or, New Reaction for, 97
————, Colour Reaction for the Detection of, 264
————, Corn Silage, Occurrence of, in, 549
————, Detection of, 407
————, Ethyl Alcohol and, Detection of, 184
————, Spirits, Detection of, in, 433
Methyl Red, Indicator, as an, 518
Methylpentoses, Rhamnose in Presence of Other, Estimation of, 410
Micro-Chemical, Aluminium, Detection of, 96, 269
————, Myrosin, Detection of, 409
———— Reactions, Anthraquinones, of Certain, 403
Micro-Elementary, Organic Substances, Quantitative, Analysis of, by F. Pregl's Methods, 280
Microscope, Nitrocelluloses, Examination with the Polarising, of, 94
Milk, Added Water, The Detection in, of, 432
——, Alcohol at Different Degrees of Concentration, Titration of, with, 317
——, Ammonia, Estimation and Significance in, of, 173
——, and, Products, The Composition of, 243
——, Boric Acid in, and in Eggs, Occurrence of, 123
——. Casein in, Volumetric Estimation of, by Means of the Tetraserum, 399
——, Condensed, the Estimation by Means of Acid Mercuric Nitrate of Cane-Sugar and Lactose in, 476
——, Electrical Conductivity of, Electrometric Determination of End-Point in Manufacture of Sweetened Condensed, 174
——, Fat Content of, and Rate of Coagulation with Rennet, 357
——, —— in, Nephelometric Method for the Determination of, 356
—— Foods, Method of Calculating the Value of, from the Point of View of, Production, 171
——, Goat's, Schardinger's Formaldehyde-Methylene Blue Reaction and Other Ferment Reactions of, 218

Milk, Hexamethylenetetramine in Wine or, Detection of, 125
——, Lactose, Polarimetric Estimation in, of, 399
——, Metabolism in, Studies in Bacterial, 494
—— Products, Milk and, The Composition of, 243
—— Samples for Analysis, Preservation of, 398
—— Serum, Abnormal Refraction of, 212
—— ——, Refraction of (Ackermann's Method), 256
——, Water, Determination of the Viscosity of, as a Means of Detecting the Addition of, 400
Millet, Hydrocyanic Acid in Feeding-Stuffs, the Estimation of, and its Occurrence in, and Guinea Corn, 430
Mineral Oil, Mixtures, Method for Calculating Viscosity of, 446
—— Oils, Asphaltic Matters in Dark, Estimation of, 178
—— Phosphates, Citric Acid, Solubility of, in, 146
—— Springs, Gaseous Hydrocarbons, Detection and Estimation of, in the Water of, 227
—— Waters, Fluorine in, 401
—— ——, Free Carbon Dioxide in, Apparatus for the Approximate Estimation of, 238
——, Sediment, Formation of in Bottled, Estimation of Silica, Iron, and Aluminium in, 126
Mineralogical, Granites and Gneisses, Quantitative, and Chemical Composition of the, 98
Molasses, Cane-Sugar in Beet, Determination of, by a Modification of the Double Polarisation (Clerget-Herzfeld) Method, Using Citric Acid, 394
——, Viscosity of Refined Syrups and, Determination of the, 220
Molecular Weight, Landsberger-Sakurai Method, Improved Apparatus for the Determination of, by the, 152
Monamino-Acids, Detection and Estimation of, 316
Monazite, Composition of, 275
—— Sand, Thorium in, Estimation of, 378
Monazites, Analysis of, 189
Monel Metal, Sample of, Analysis of a, 561
Montana Waxes, Montanin and, 93
Montanin and Montana Waxes, 93
Morphine, Acid Opium Solutions, Estimation of, in, 31
——, Uranium Salts, Detection of, and Phenols by Means of, 438
Moslinger's Barium Chloride Method, Lactic Acid in Wine, Estimation by, of, 397
Mowrah Fat, Animal and Vegetable Fats, Unsaponifiable Constituents of, and the Detection of this Fat in, 310
——, Shea Butter, Unsaponifiable Constituents of, and their Relation to Those of, 440
Musts, Organic Phosphoric Acid Compounds in Concentrated, and Wines, 398
Muta-Rotation, the Double Polarisation (Clerget-Herzfeld) Method of Determining Cane-Sugar, as a Source of Error in, 258
Mutton and Beef Fats, Lard, Detection by Bömer's Method of, in, 253
Myrosin, Microchemical Detection of, 409

Nephelometer, Acetone, Estimation of Small Quantities of, in the, 178
——, Analysis, New, and its Use in, 283
Nephelometric Method, Milk, for the Determination of Fat in, 356
Nephelometry, Nucleases, in the Study of, 359
New South Wales, Cyanogenetic Plants of, 442

Nickel, Cobalt, Iron, and Copper, Colorimetric Estimation of, 326
——, Colorimetric Estimation of, 327
—— Cooking-Vessels, 417
——, Dimethylglyoxime, Estimation of, by Means of, 375
——, Electrolytic Separation of, 454
——, Fats, Detection of, in, 254
Nicotine, Ammonia, Estimation of, in the Presence of, 42
——, Silicotungstate, Estimation of, as, 43
Nitrates, Chlorates, and Peroxides, Reduction Method of Analysing, 188
——, Formic Acid, Reaction of, with, 233
——, Nitrites, Colorimetric Method for Detecting and Estimating, and, 276
——, Nitrocelluloses and, Estimation of Nitrogen in, 276
Nitric Acid, Platinum Vessels, Resistance to Hot, of, 382
——, Rainfall, Influence of Weather Conditions upon the Amounts of, and of Nitrous Acid in the, 501
Nitrites, Free Chlorine, Effect of Ferric Salts and, on the *o*-Tolidine and Starch Iodide Tests for, 454
——, Fresenius's Method for Determining Small Quantities of, On, and its Sensitiveness Compared with the *m*-Phenylene-Diamine Reaction, 350
——, Gravimetric Estimation of, 145
——, Nitrates, Colorimetric Method for Detecting and Estimating, and, 276
Nitrocellulose Manufacture, Waste Acids from, Analysis of, 183
Nitrocelluloses, Nitrates and, Estimation of Nitrogen in, 276
——, Polarising Microscope, Examination of, with the, 94
——, Sulphuric Acid, Estimation in, of, 45
Nitro-Compounds, Nitrogen in, Estimation by Kjeldahl's Method of, 93
——, Titanium Chloride, Volumetric Estimation of, and Nitroso-Compounds by Means of, 134
Nitrogen, Alkyl in Combination with, Estimation of, 226
——, Bomb Calorimeter, Oxidation of Sulphur Compounds of Coal, and of, in the, and the Correction to be Applied in Determining the Heating Value of Coal, 563
——, Calcium Nitrate and Calcium Cyanamide, Estimation of, in Mixtures of, 329
—— Generator, 421
——, Guncotton, Determination by Means of the Nitrometer of, in, 409
——, Kjeldahl Method of Estimating, Modifications of the, 227
——, Nitrates and Nitrocelluloses, Estimation of, in, 276
——, Nitro-Compounds, Estimation of, in, by Kjeldahl's Method, 93
—— Peroxide, Ozone, and Hydrogen Peroxide at High Dilutions, Estimation and Distinction of, 189
——, Rain and Snow, and Chlorine in, 190
——, Vegetable, Yeast, and Meat Extracts, Distribution of, in, 444
Nitrogenous Constituents, Hops, of, 492
Nitrometer, Guncotton, Determination by Means of the, of Nitrogen in, 409
α-Nitroso-β-Naphthol, Palladium, Estimation of, by Means of, and Separation of Palladium from Copper and Iron, 55
Nitroso-Compounds and, Volumetric Estimation of, by Means of Titanium Chloride, 134
Nitrosophenylhydroxylamine, Titanium from Iron, Use of Ammonium Salt of ("Cupferron"), in Quantitative Separation of, 332
——, Zirconium from Iron and Aluminium with the Aid of the Ammonium Salt of ("Cupferron"), Separation of, 565
Nitrous Acid, Rainfall, Influence of Weather Conditions upon the Amounts of Nitric Acid and of, in the, 501

Noble Metals, Cupellation, Loss of, on, 145
Nomenclature, Alloys, Summary of First Report of Committee on the, of, 286
Non-Uniformity, Temperatures, of Drying-Oven, 457
Nucleases, Nephelometry in the Study of, 359
Nuts, Cohune, Fat of, 396
Nux Vomica, Preparations of, Estimation of the Alkaloids, Application to, 122

OBITUARY NOTICE:
Bell, James Carter, A.R.S.M., 1
Occluded Salts, Silica for, Correcting, 191
Ocymum Pilosum, Roxb., Essential Oil of, 438
Odour, "Urinod," the Cause of the Characteristic, of Urine, 554
Oil Asphalts, Natural and, Differentiation of, 362
—, Black Sage, Investigations on, of, 559
— Fuel, Crude Petroleum, and Similar Substances, Two Rapid Methods of Estimating Water in, 529
— Seeds, Oils and, New or Little-Known, 134
Oils, Acetyl Value of, Fats, Etc., Estimation of the, 362
— and Fats, The Fachini-Dorta Method of [Approximately] Separating Liquid from Solid Fatty Acids in, 389
—, *Coniferæ*, of the, Leaf and Twig, and Bark, of White Fir, 558
—, Digitonin Method, Estimation of the Unsaponifiable Constituents of, and Fats, and of Lanolin by Means of the, 310
—, Insoluble Bromide Value of, Note on the, 388
—, ————, The, and its Determination, 297
—, Ricinoleic Acid in, Estimation of, 410
—, Rosin in Varnishes, and Soaps, Estimation of, 228
—, Volatility or Oxidisability of, Apparatus for Testing the, 334
—, Viscosity of, 185
Oleic Acid, Halogens, Action on, of, and Estimation of the Iodine Value of Fats, 311
————, Rancidity of Olive Oil and Oxidation of, in Presence of Sunlight, 255
Olive Oil, Oxidation of Oleic Acid and Rancidity of, in Presence of Sunlight, 255
Open-Hearth Steel, Oxygen Content of, 504
Opium Solutions, Morphine in Acid, Estimation of, 31
Ore, Bog Iron, Destruction of Organic Matter in, 372
Ores, Iron in Iron, Estimation of, 144
—, Radium from Australian, Extraction of, 455
ORGANIC ANALYSIS, ABSTRACTS, 40, 91, 131, 178, 225, 264, 319, 362, 403, 445, 496, 555
Organic Compounds, Halogens, Estimation in, of, 367
————, Oxygen, Estimation in, of, 135
————, Trichloracetic Acid, Colour Reactions of, with, 410
—— Fluids, Collargol, Estimation of Silver in, and, 278
—— Matter, Bog Iron Ore, Destruction of, in, 372
—— Phosphoric Acid Compounds, Concentrated Musts and Wines, in, 398
—— Residue, Crude Glycerol by the International Standard Methods, Estimation of, 34
—— Substances, Arsenic, Estimation in, of, 442
————, Boric Acid in, Method of Estimating Very Small Quantities of, 96
————, F. Pregl's Methods, Quantitative Micro-Elementary Analysis of, by, 280
Iodine, Estimation in, of, 367
Tissues, Amino-Nitrogen in, Estimation of, 175

Orthotolidine, Free Chlorine, as Reagent for Colorimetric Estimation of Small Quantities of, 46
Oxalate, Calcium as, Gravimetric Estimation of, 50
Oxidation, Chromium, Estimation of, by, in Alkaline, 143
Oxides, Lead, Analysis of Higher, of, 328
Oxidisability, Oils, Apparatus for Testing the Volatility or, of, 334
Oxidising Substances, Diphenylamine as a Reagent for; Use of Diphenylbenzidine in Place of, 320
Oxycholesterol, Cholesterol and, Estimation of by the Method of Autenrieth and Funk, 129
Oxydases, Guaiacum Resin as a Reagent for the Detection of, and of Minute Traces of Copper, 39
Oxygen Absorption, Linseed Oil, Gravimetric Estimation of the, of, during Drying, 92
——— Content, Open-Hearth Steel, of, 504
  Organic Compounds, Estimation of, 135
  Soaps and Soap Powders Containing Per-Salts, Apparatus for the Estimation of Active, in, 228
  Water, Estimation of Dissolved, in, 55
  ———, Field Method for Determining Dissolved, in, 234
  ———, Solubility of Atmospheric, in, 55
Oxyproteic Acid Fraction, Urine, Quantitative Estimation of the, in, 361
Ozone, Nitrogen Peroxide, Hydrogen Peroxide, and, at High Dilutions, Estimation and Distinction of, 189

Paint Vapours, Composition of, 265
Paints, Steel, Tests of, for, Subjected to Alternate Exposure to Air and Fresh Water, 54
Palestine, Thermal Springs in, Analyses of Waters from, 102
Palladium, α-Nitroso-β-Naphthol, Estimation of, by Means of and Separation of, from Copper and Iron, 55
Pancreas, Amylolytic Diastase of, Assay of the, 318
——— Preparations, Proteolytic Activity of, Comparison of Methods for the Determination of the, 551
Papain, Commercial, and its Analysis, 444
Paper, Dry and Wet Strengths of, and Yarns, 322
———, Drying of, for Test Purposes, Apparatus for the, 508
Paprika, Extracted, Detection of, 174
"Paraffin, Shrewsbury's Reaction for," Note on, 43, 296
——— Wax, Petroleum Oil, Analysis of, and, 43, 266
Pemberton-Kilgore Method, Phosphoric Acid, Study of, for Determination of, 100
Pentosuria, Urine, Sugars in, in Cases of, 40
Pentoses, Carbohydrates, Methods of Estimation of. III. The Cupric-Reducing Power of the—Xylose and Arabinose, 555
Pepsin, Valuation of, 130
Peptides, Urine, Detection of, in, by the *p*-Cresol Tyrosinase Reaction, 555
Perchlorates, Acid Radicals of Chlorides, Chlorates, Bromides, Bromates, and Fluorides, Application of Sodium Paratungstate and the Blowpipe Flame to the Estimation of the, 188
Periodate, Iodate and Iodide, Estimation of, in Presence of, 56
Permanganate Titration, Iron for, Electrolytic Reduction of, 328
———————, Iron in Presence of Chlorides, of, 415
Peroxides, Nitrates, Chlorates, and, Reduction Method of Analysing, 188
Peroxydase Reaction, Delicacy of the, 402

Per-Salts, Active Oxygen in Soaps and Soap Powders Containing, Apparatus for the Estimation of, 228
Peru Balsam, 400
Petroleum Distillates, Fluorescence of, 446
——— Oil, Paraffin Wax and, Analysis of, 43, 266
———, Water in Crude, Oil-Fuel, and Similar Substances, Two Rapid Methods of Estimating, 529
Petroleums, California, Specific Heat of, 499
Pharmaceutical Chemical Standards, 439
Phenacetin, Acetanilide and, in Admixture, Estimation of, 433
Phenol, Hexamethylenetetramine and Formaldehyde, Determination of, in the Presence of, 266
Phenols, Morphine and, Detection by Means of Uranium Salts of, 438
m-Phenylene-Diamine Reaction, Nitrites, On Fresenius's Method for Determining Small Quantities of, and its Sensitiveness compared with the, 350
Phlorin, Phlorrhizin Alone and in Presence of, Detection of Minute Quantities of, 410
Phlorrhizin, Phlorin, Detection of Minute Quantities of, Alone and in Presence of, 410
Phosphate, Titanium, Estimation of, as, 279
Phosphates, Citric Acid, Solubility of Mineral, in, 146
———, Food Materials, Volumetric Estimation of, in the Ash of, 234
Phosphomolybdic Acid, Saffron, Detection of Adulterants in, by Means of, 219
Phosphoric Acid, Basic Slag, Estimation of Citric Acid Soluble, in, by the Citrate Method and Lorenz's Method, 377
———, Basic Slag, Estimation of Citric Acid Soluble, in, 377
——— Compounds, Musts and Wines, Organic, in Concentrated, 398
———, in the Presence of, Esters, Estimation of, 559
———, Iron, Determination in Presence of, of, 416
———, Pemberton-Kilgore Method for Determination of, Study of, 100
———, Sodium Citrate, Use of, for Determination of Reverted, 276
Photographic Developers, Synthetic Drugs, New Remedies, etc., Index List of Trade Names of, 533
Physical Constants, Isoprene, of, 406
Physico-Chemical, Sulphates, Estimation of, 191
———, Volumetric Method, New, 330
Physiological Liquids, Blood and Other, Estimation of Carbohydrates and Lactic Acid in, 551
Phthaleïns, Indicators, Properties of the, as, 136
Phytosteryl Acetate Test, Bömer's, and Polenske's Distillation Method, Detection of Coconut Oil in Butter Fat by Means of, 254
Phytin, Estimation of, Volumetric Method for the, 560
Phytosterol, Animal Fats, Detection of, in, 84
Picrate Colorimetric Method, Carbohydrates, for the Estimation of, 180
Picric Acid, Iodimetric and Acidimetric Titrations, as a Standardising Substance for, 267
———, Urine, Detection of, in, 224
Pig-Iron, Sulphur in, and Steels, Colorimetric Estimation of, 273
Pills, Camphor, Estimation in Tablets, and, of, 353
Pimento, Essential Oils and Essential Oil Products, Hydrogen Value of Some. I. Oils of Sassafras, Anise, Fennel, Clove, and, 557
Pine Woods, Fractional Distillation of Mixed, 48
Pines, Cuban and Long-Leaf, Leaf and Twig Oils of, and Cone Oil of Long-Leaf Pine, 498
Pipettometer, 509
Pitch, Softening-Point of, Determination of the, 136, 321

Pitot-Tubes, Gas Velocities, for Measurement of, 456
Plant Analysis, Lævulosans, The Hydrolysis of, and its Application in, II. and III., 88
—— Material, Estimation of Starch in, 312
—— Tissues, Quantitative Extraction of Diastases from, 260
Plants, Carbon Dioxide and, 260
——, Cement, Estimation of the Dust Fall in the Neighbourhood of, 372
——, Medicinal, and Drugs, Use of Carbon Tetrachloride in the Extraction and Estimation of the Active Principles of, 255
——, New South Wales, Cyanogenetic, of, 442
——, Volatile Oils, Estimation of, in Aromatic, Drugs, and Spices by the Tailameter, 35
Platinum-Iridium Alloys, The Determination of Iridium in, 340
—— Vessels, Resistance to Hot Nitric Acid of, 382
—— Ware, Thermo-Electric Method for the Determination of the Purity of, 381
Podophyllum, Resin, Estimation of, 439
Poisoning, Lead, Estimation of Small Quantities of Lead, particularly in Urine, in Cases of, 495
Poisonous Bran, 215
Polarimetric Estimation, Lactose in Milk, of, 399
Polarisation Method, Cane-Sugar, Constants for the Double [Clerget-Herzfeld], of Determining, 86
Polenske's and Bömer's Methods, Lard, Detection of Beef Fat in, by, 216
—— Distillation Method, Detection of Coconut Oil in Butter Fat by Means of, and Bömer's Phytosteryl Acetate Test, 254
Polypeptide-Nitrogen, Barley, Malt, and Beer, Estimation of Amino-Acid and, in, by Titration with Acid after Treatment with Formaldehyde, 257
Portland Cement, Blast-Furnace Slag, Estimation in, of, 187
Potash, Small Quantities of, Volumetric Estimation of, 417
Potassium, Potassium Hydrogen Tartrate, Estimation of, as, 330
—— Xanthate, Titration of, 95
Potato Starch, Finishing Purposes, Rapid Testing of, for, 94
Poultry, Cattle and, Food: "Artificial Preparation." Worcestershire County Council *v.* Notley Brothers (Law Report), 287
Powder, Black, Examination of, 267
——, Liquorice, The Composition and Analysis of Compound, 160
Precious Metal, Silver and Base Metal in, Bullion, Determination of, 502
Precipitators, Instantaneous Thermostat and Smoke and Fume Monitors, and Recorders, 567
F. Pregl's Methods, Organic Substances, Quantitative Micro-Elementary Analysis by, 280
Pregnancy, Early Stages of, Determination of, 177
Preservation, Milk Samples, of, for Analysis, 398
Preservatives, Urine, Comparison of Various, for, 177
——, Wood (Creosote Oil), Report of the Committee on, 497
Pressure Gauge, New Differential, 60
Pressures, Vapour, New Method for the Determination of, and an Examination of a Source of Error in Certain Dynamical Methods, 511
Printing Inks, Analysis, and their, 450
Proprietary Foods, Local Government Board, Reports to the. I. On the Use of, for Infant Feeding. II. On the Analysis and Composition of Some, for Infants, 464
Protein, Lactic Acid, Estimation of, in Liquids Containing, 133
Proteins, Herzig and Meyer's Reaction Applied to, and Amino-Acids, 317
——, Urine, Centrifugal Method for Estimating, in, 40
——, Wheat Flour, Estimation of the, in, 550

Proteins, Wheat Flour, Quantitative Estimation of the, Soluble in Salt Solutions in, 262
Proteolytic Activity, Pancreas Preparations, Comparison of Methods for the Determination of the, of, 551
Proximate, Analysis of Wheat, 441
Pulp, Wood for, Chemical Valuation of, 451
Pycnometer Spindle, Alcohol, a New Instrument for Use in the Estimation of, 284
Pyridine, Hydroxyl Groups, as a Solvent in the Estimation of, by Means of Alkyl Magnesium Halides, 186
Pyrophoric Alloys, Cerium and Iron, Analysis of, of, 415
Pyruvic Acid, Estimation of, 137

Quinine, Strychnine, the Estimation of, in the Presence of, 81

Radicles, Cacao Beans, Composition of the, of, 308
Radio-Active Substances, Radium in, Estimation of, 57
Radio-Activity, Spring Waters, Fontactoscope for Measuring the, of, 423
——————, Springs, Measurement of, of, 277
Radio-Elements, Indicators in Analytical Chemistry, as, 57
Radium, Australian Ores, Extraction of, from, 455
———— Emanation, Altitude and Meteorological Conditions, Quantitative Determination of, in the Atmosphere, and its Variation with, 418
————, Emanation Method, Estimation of, by the, 58
————, Radio-Active Substances, Estimation of, in, 57
Rain, Nitrogen and Chlorine in, and Snow, 190
Rainfall, Nitric Acid and of Nitrous Acid in the, Influence of Weather Conditions upon the Amouuts of, 501
(Rare Earths, Fractionation of), Dimethyl Phosphates of the Rare Earths, 146
——————, Hydroxides, Errors in the Estimation of, as, 330
Rare Metals, Tobacco Ash, Titanium and, in, 43
Rational Analysis, Clays, of, 499
Reagents, Fluorine, Occurrence of, in, 231
————, Lead, Sensitiveness of Various, for, 374
Recorders, Instantaneous Thermostat and Smoke Fume Monitors, Precipitators, and, 567
Red Lead, Analysis and Composition of, 375
Reducing Sugars, Cane-Sugar, Determination of a Small Amount of, in the Presence of a Large Proportion of, 45
Reductase (Fermentation-Reductase) Test, 224
Reduction Tests, Dextrose, Influence of Metals Present in Urine on the Velocity of the, for, 131
Reductor, Titanium, and Chromium, Volumetric Estimation of, by Means of a Modified, 419
Refraction, Milk Serum, Abnormal, of, 212
Refractometer, Sugar, 59
Remedies, Trade Names of Synthetic Drugs, New, Photographic Developers, etc., Index List of, 533
Rennet, Milk, Fat Content of, and Rate of Coagulation with, 357
Report, Cocoa and Cocoa Products, on, 123
————, Government Laboratory, of the Government Chemist upon the Work of the, for the Year ended March 31, 1914, with Appendices, 458
————, Government, on the Work of the Inspectors of Foods for the Year 1912-13, 195
————, Metropolitan Water Board. Tenth Research, 514

Report, Metropolitan Water Board. Eleventh Annual, for the Year ended March 31, 1914, 512
——, Wood Preservatives (Creosote Oil), of the Committee on, 497
Reports, Local Government Board, to the. I. On the Use of Proprietary Foods for Infant Feeding. II. On the Analysis and Composition of Some Proprietary Foods for Infants, 464
Resin, Guaiacum, as a Reagent for the Detection of Oxydases and of Minute Traces of Copper, 39
——, Podophyllum, Estimation of, 439
Resins, Tobacco, 220
Reverted Phosphoric Acid, Sodium Citrate for Determination of, Use of, 276
Reviews of Books:
  Agricultural Analysis, Principles and Practice of, 423
  Alkali, Sulphuric Acid and, the Manufacture of, with the Collateral Branches, 200
  Alkaloids, Plant, The, 104
  Analysis, Quantitative Chemical, A Textbook of, 240
  ——, Quantitative Inorganic, A Treatise on, with Special Reference to the, of Clays, Silicates, and Related Minerals, 155
  Bacteriology, Water, Elements of, 157
  Bases, Natural, The Simpler, 574
  Beurre, Fraudes dans le Commerce du, Recueil de Législations Beurrières et Margarinières Concernant la Répression des, 474
  Catalyse, Chimie Organique, La, en, 65
  Chimie Organique, Principes d'Analyse et de Synthèse en, 383
  Disinfection, Disinfectants, and, 66
  Drinking-Water, Microscopy of, The, 472
  Fæces, Children and Adults, The, of, 242
  Fuel, Gaseous, Solid, and Liquid, 575
  Hexite and Pentite, Stereo-Chemical Theory of General Application, the Exposition of a, Theory of a, The Silicates in Chemistry and Commerce, Including, 201
  Institute of Chemistry, History of the, 1877-1914, 570
  Organic Chemistry, Students, for Advanced. Vol. II., 106
  Reagenzien-Verzeichnis, 65
  Sewage, Water Purification and, Disposal, 66
  Water, Clean, and How to Get It, 473
Rhamnose, Methylpentoses, Estimation of, in Presence of Other, 410
Rice, "Dead," Composition of, 550
—— Starch, Liquefaction of, Fractional, 440
Ricinoleic Acid, Oils, Estimation of, in, 410
Roads, Tar Treatment of, Revision of Road Board Specifications, 267
Roe, the Soft, and Hard Roe of Fishes, Composition of, 261
Rose Oil, Composition and Adulteration of, 493
Rosin Size, Examination of, 139
——, Sizings, Identification of, and Glue, 411
——, Varnishes, Oils, and Soaps, Estimation of, in, 228
Rotating Cathode, Electrolysis with the, Apparatus for Estimation of Small Quantities by, 334
——, Electrolytic Analysis, and Anode for Rapid, 334
Rotation, Lactose, Influence of Ammonium Sulphate on the, of, 133
Rubber, Caoutchouc in Crude, Direct Estimation of, 179
——, ——, Estimation of, 138
—— Solutions, Viscosity of, 322

Rubber Sulphur, The Estimation in, of, 74
———, Thirty per Cent. *Hevea*, Insulating Compound, Analytical Procedure for, 137
Rums, Composition of, 401
Ruthenium Red, Sinews, Treatment of, particularly by the Application of, and the Mallory Connective Tissue Stain, 89
Rye Flour, Wheat Flour, Detection of Small Quantities of, in, 400

Saccharin, Detection and Estimation of, 401
————, Extraction by Means of Immiscible Solvents from Point of View of Distribution Coefficients, Study of Methods of. I.: (Analysis of Vanilla Extracts and Extraction of Acetanilide, Saccharin, and Caffeine), 354
————, Foods, Estimation of, in, 86
Saffron, Adulterants in, Detection of, by Means of Phosphomolybdic Acid, 219
———, Analysis of, 312
———, Magnesium Sulphate in, Detection of, 219
Sage, Black, Investigations on Oil of, 559
Salicylic Acid, Jams, Estimation of, in, 219
Saline Matter, Wet Salted East India Goat Skins, Analysis of the, Adhering to the Flesh Side of, 402
Salt Solutions, Proteins Soluble in, in Wheat Flour, Quantitative Estimation of, 262
Salts, Alkaloids and Alkaloid, Solvents for, 31
——, Aporeine and its, 252
——, Bromine Associated with Chlorine in Haloid, Use of Telluric Acid in Estimation of, 331
——, Ferric, New Method of Titrating, 189
——, Ferricyanide in Presence of Ferric, and Cyanides, Volumetric Estimation of, 366
——, Lead, Action of Sodium Hydrogen Carbonate Solutions on Difficultly Soluble, 143
——, Morphine and Phenols, Detection of, by Means of Uranium, 438
——, *o*-Tolidine and Starch Iodide Tests for Free Chlorine, Effect of Ferric, and Nitrites on the, 454
——, Silica, Correcting for Occluded, 191
——, Tanning Materials, Application of Tungsten, to the Analysis of, 507
Sand, Monazite, Estimation of Thorium in, 378
Saponin, Aerated Waters, Detection of, in, 440
———, Detection of, 175
Sassafras. I.: Oils of, Anise, Fennel, Clove, and Pimento. Hydrogen Value of Some Essential Oils and Essential Oil Products, 557
Schardinger's Formaldehyde-Methylene Blue Reaction and Other Ferment Reactions of Goat's Milk, 218
Schiff's Reagent, Formaldehyde and Formaldehyde Compounds, Detection of, by Means of, Containing Hydrochloric Acid, 172
Seal, Aeration in De-Aerated Liquids, New, for Prevention of, 509
Sediment, Bottled Mineral Waters, Formation of, in. Estimation of Silica, Iron, and Aluminium in Mineral Waters, 126
Seeds, Cacao, 171
—, Castor, The Detection of, 292
—, Oil, and Oils, New or Little-Known, 134
Selenious Acid, Sulphuric Acid, Detection of Small Quantities of, in, 419
——————, Very Small Quantities of, Detection and Estimation of, 100
Selenium, Gravimetric Estimation of, 235
Semicarbazide, Halogens and Halogen Oxyacids, Estimation of, by its Interaction with, 323

Senna Leaves, Constituents of, 35
Serum, Blood, Estimation of Chlorine in, 223
———, Milk, Abnormal Refraction of, 212
———, ———, Refraction of (Ackermann's Method), 256
Sesame Oil, Margarine Containing Colouring Substances, Detection of, in, 86
Shaking Apparatus, 284
"Sharps," Notes on, 251
Shea Butter, Mowrah Fat, Unsaponifiable Constituents of, and their Relation to Those of, 440
Shellac, Bleached, Examination of, 139, 411
"Shrewsbury's Reaction, Paraffin," for, 43
————————, ————, Note on, for, 296
Sikes's Hydrometer, Proof Strengths of Spirits at Temperatures from 40° F. to 100° F. by, Method by which were Calculated the Corrected Sikes's Tables in Use in India, for Determining the, 532
Silage, Methyl Alcohol, Occurrence in Corn, of, 549
Silica, Dehydration and Separation of, 190
———, Mineral Waters, Estimation of, Iron, and Aluminium in. Formation of Sediment in Bottled Mineral Waters, 126
———, Occluded Salts, Correcting, for, 191
———, Solubility of, and Rendering, Insoluble in Acid Solutions, 191
Silicates, Melting-Points of, Determination of, 236
————, Soap, Test for, in, 550
Silicic Acids, Estimation of, 455
"Silicol," Hydrogen, Analysis of, and Other Silicon Compounds Used for the Production of, 147
Silicon, Alloys, Analysis of Copper, Tin, and, 500
——— Compounds, "Silicol" and Other, Used for the Production of Hydrogen, Analysis of, 147
Silicotic, Lungs, Ash of, 359
Silicotungstate, Nicotine as, Estimation of, 43
Silver, Collargol and Organic Fluids, Estimation of, in, 278
———, Colloidal, Estimation of, in, 279
——— -Plated Copper Gauze Electrode in Zinc Determination, 382
———, Precious Metal Bullion, Determination of, and Base Metal in, 502
Sinews, Ruthenium Red and the Mallory Connective Tissue Stain, Treatment of, particularly by the Application of, 89
Size Rosin, Examination of, 139
Sizings, Rosin and Glue, Identification of, 411
Skins, Goat, Analysis of the Saline Matter Adhering to the Flesh Side of Wet Salted East India, 402
Slag, Citric Acid Soluble Phosphoric Acid, Estimation in Basic, of, 377
——, Citric Acid Soluble Phosphoric Acid in Basic, Estimation of, by the Citrate Method and Lorenz's Method, 377
——, Portland Cement, Estimation of Blast-Furnace, in, 187
Smoke and Fume Monitors, Instantaneous Thermostat and, Precipitators, and Recorders, 567
Snow, Rain and, Nitrogen and Chlorine in, 190
Soap, Lathering Capacity of, 312
Lyes, Technical Glycerines and, Estimation of Glycerol in, 181
——— Powders, Active Oxygen in Soaps and, Containing Per-Salts, Apparatus for the Estimation of, 228
——— Silicates in, Test for, 550
Soaps, Fats and, Method of Estimating Colophony in, 181

Soaps, Fatty Acids, Estimation in, of, 311
——, Free Alkali, Estimation in, of, 140
——, Free Alkali in Commercial Soda and, Estimation of, by the Barium Chloride Method, 229
——, Rosin in Varnishes, Oils, and, Estimation of, 228
SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS, PROCEEDINGS OF, 4, 67, 108, 159, 203, 243, 291, 340, 385, 425, 475, 517
Soda, Free Alkali in Commercial, and Soaps, Estimation of, by the Barium Chloride Method, 229
Sodium Citrate, Reverted Phosphoric Acid, Use of, for Determination of, 276
Sodium Hydrogen Carbonate Solutions, Lead Salts, Action of, on Difficultly Soluble, 143
—— Hypoiodite, Aldoses, Estimation of (Part I.), Action of Neutral, 556
—— -Nitro-Prusside Reaction, Urine, Violet, of, 264
—— Paratungstate, Acid Radicals in Chlorides, Chlorates, Perchlorates, Bromides, Bromates, and Fluorides, Application of, and the Blowpipe Flame to the, Estimation of the, 188
Softening-Point, Pitch, Determination of the, of, 136, 321
Soil Carbonates, Determination of, 562
—— Extracts, Estimation of Carbon in Soils and, 449
——, —— of Phosphates in, 329
——, Lime Requirements of, Determination of, 450
Soils, Arsenic as a Normal Constituent in, 49
——, Capillary Lift of, Measuring the, 318
——, Carbon, Estimation in, and Soil Extracts of, 449
——, Heavy Clay, Determination of Humus in, 132
——, Lime Requirement of, Estimation of the, 90
Solidifying Points of Mixtures of Fatty Acids, Melting and, and Use of these Points to Determine Composition of Such Mixtures, 448
Soluble Substances, Analysis of Adhesive, 252
Solubilities, High Temperatures, New Method for Determining, at, 335
Solvent, Varnish, Quantitative Determination of Body and, in, 560
Solvents, Alkaloids and Alkaloid Salts, of, 31
——, Coal, Action of, on, 406
——, ——, for, 320
——, Extraction Apparatus Specially Designed for, of High Specific Gravity, 240
Spectroscopic Analysis, Absorption Tube for, Improved, 153
Spices, Volatile Oils, Estimation of, in Aromatic Plants, Drugs, and, by the Tailameter, 35
Spirits, Methyl Alcohol, Detection in, of, 433
——, Proof Strengths of, at Temperatures from 40° F. to 100° F. by Sikes's Hydrometer, Method by which were Calculated the Corrected Sikes's Tables in Use in India for Determining the, 532
Spring Waters, Radio-Activity of, Fontactoscope for Measuring the, 423
Springs, Radio-Activity of, Measurement of, 277
Stabilised Cacao, Analysis of, 434
Stag's Fat, Characteristics of, 32
Standard Methods, Crude Glycerol, Analysis of, by the International, 1911. Estimation of Organic Residue, 34
—— Tests, Lubricants, for, 557
Standardisation, Volumetric Arsenite Solutions, of, 271
Standardising Substance, Iodimetric and Acidimetric Titrations, Picric Acid as a, for, 267

Standards, Pharmaceutical Chemical, 439
Starch, Indicator, Preparation of Dry, Soluble in Cold Water, for Use as an, 451
——— Iodide, *o*-Tolidine and, Tests for Free Chlorine, Effect of Ferric Salts and Nitrites on the, 454
———, Plant Material, Estimation of, in, 312
———, Rice, Fractional Liquefaction of, 440
Starches, Variety of, 314
Steam Generator, 152
Steel, Carbon in, and Iron, Determination by the Barium Carbonate Titration Method of, 373
———, ———, by the Direct Combustion Method, Determination of, 560
———, Chromium and Manganese, Estimation in Iron and, of, 454
———, Cobalt in, Estimation of, 271
———, Open-Hearth, Oxygen Content of, 504
———, Paints for, Subjected to Alternate Exposure to Air and Fresh Water, Tests of, 54
Steels, Cobalt in High-Speed, Estimation of, 503
———, Sulphur in Pig-Iron and, Colorimetric Estimation of, 273
Still, Fractional Distillation, for Exact Measurement of Boiling-Point during, 456
Stirring Apparatus, 422
Strychnine, Brucine, Estimation of, in Presence of, 394
———, Manganese Carbonate, Identification of Traces of, by Means of, 357
———, Quinine, The Estimation of, in the Presence of, 81
Sublimation, Quantitative, 194
———, Temperature of, 153
Substances, Finely Powdered, Estimation of Crude Fibre in, 218
Succinic, Malic and, Acids, Colour Reaction of, 186
Suction, Device to Assist Filtration by, 508
Sugar, Blood, Estimation of, in, 90
———, ———, Micro-Estimation of, in, 90
———, Cane, Constants for the Double Polarisation (Clerget-Herzfeld) Method of Determining, 86
———, ———, Determination of a Small Amount of Reducing Sugars in the Presence of a Large Proportion of, 45
———, ———, in Beet Molasses, Determination of, by a Modification of the Double Polarisation (Clerget-Herzfeld) Method, using Citric Acid, 394
———, ———, Inversion of, by Acids in Water-Alcohol Solutions, 323
———, ———, Muta-Rotation as a Source of Error in the Double Polarisation (Clerget-Herzfeld), Method of Determining, 258
———, Honey and Other Substances Rich in, Estimation of Sulphate in, 259
———, Lactose, Estimation of Cane, in the Presence of, 363
———, Refractometer, 59
———, Urine, Simple Colorimetric Method for the Estimation of, in, 224
Sugars, Gingerbread and Similar Products, Separation and Estimation of, in, 358
———, Lævulose, Detection and Estimation in Presence of Other, of, 323
———, Reducing, Determination of a Small Amount of, in the Presence of a Large Proportion of Cane Sugar, 45
———, Urine, in, in Cases of Pentosuria, 40
Sulphate, Honey and Other Substances Rich in Sugar, Estimation of, in, 259
Sulphates, Flour, Note on the Presence of, in, 429
———, ———, Notes on: 1. Acidity of Flour; 2. Natural and Artificial Bleaching of Flour; 3. Sulphates and Lime in Flour, 519
———, Physico-Chemical Estimation of, 191
———, Water of Crystallisation, Estimation of, in, 193

Sulphide of Antimony, Golden, Analysis of, 186
Sulphite, Thiosulphate, Estimation in Presence of, of, 101
Sulphites, Thiosulphates, Detection and Approximate Estimation in, of Traces of, 147
Sulphur, Commercial Benzene, Estimation of Total, in, 140
——— Compounds, Oxidation of, of, and of Nitrogen in the Bomb Calorimeter, and the Correction to be Applied in Determining the Heating Value of Coal, 563
———————, Urine, Rapid Estimation of Different, in, 101
——— Dioxide, Air, Estimation of Minute Amounts of, in, 376
——————, Gelatin, in, 87
-, Hydrocarbons, Separation by Means of Liquid, of, 321
———, India-Rubber, Estimation of Total, in, 368
———, Methoxyl in Compounds Containing, Estimation of, 265
———, Pig-Iron and Steels, Colorimetric Estimation of, in, 273
———, Rubber, the Estimation of, in, 74
Sulphuretted Hydrogen, Graphite, from Artificial, 67
Sulphuric Acid, Blende and Galena, Velocity of Solution in Dilute, of, 452
—————, Free Sulphurous Acid in Presence of Thiosulphate or, Estimation of, 279
—————, Nitrocelluloses, Estimation of, in, 45
--, Selenious Acid, Detection in, of Small Quantities of, 419
———— Anhydride, Chlorine, The Chief Cause of the Loss of, and of, by Incinerating Substances Containing these Constituents, 425
————, Hydrochloric and, Acids, Estimation of Arsenic in, 370
————, Sulphurous and, Acids in the Atmosphere, On Damage Caused to Vegetation by, 203
Sulphurous Acid, Thiosulphate or Sulphuric Acid, Estimation of Free, in Presence of, 279
——————, Volumetric Estimation of, 331
——————, Wine, Estimation of Total, in, 87
Sunlight, Rancidity of Olive Oil and Oxidation of Oleic Acid in Presence of, 255
Synthetic Caoutchouc, Isoprene, Note on, from, 179
———— Drugs, Trade Names of, New Remedies, Photographic Developers, Etc., Index List of, 533
Syrups, Refined, and Molasses, Determination of the Viscosity of, 220

Tablets, Camphor, Estimation in, and Pills of, 353
———, Mercuric Iodide in, Estimation of, 493
Tailameter, Volatile Oils, Estimation in Aromatic Plants, Drugs, and Spices of, by the, 35
Tannin, Cider, Estimation of, in, 127
——— Reaction, Anthocyanenes, New, and its Relation to the, 412
Tanning Materials, Tungsten Salts, Application to the Analysis of, of, 507
Tar Roads, Treatment of. Revision of Road Board Specifications, 267
—— Products, Free Carbon in, Estimation of So-Called, 364
—— Water-Gas, Composition of, 369
Tars, Refined, Relation between the Melting-Point and Viscosity of, 369
Tasmanian Eucalpyts, Correlation between the Specific Characters of the, and Australian, 435
Teichmann's Test, Blood, Modification of, for, 128, 315
Telluric Acid, Bromine Associated with Chlorine in Haloid Salts, Use of, in Estimation of, 331
Tellurium Dioxide, Combustion Analyses, for, 192

Tellurium, Gravimetric Estimation of, 379
Temperature, Corrections to be Applied to the Specific Gravities of Malt Worts and Beers, 256
————, Fusing, Relation of Composition of Ash of Coal to its, 445
Temperature of Sublimation, 153
————, Temperatures Very Close to Room, Thermo-Regulator for Thermostats at, 568
Temperatures, Drying-Oven, Non-Uniformity of, 457
————, Low, Gas Analysis by Fractional Distillation at, 414
————, Solubilities at High, New Method for Determining, 335
Test Purposes, Paper for, Apparatus for the Drying of, 508
Tetraserum, Casein in Milk, Volumetric Estimation of, by Means of the, 399
Theobromine, Mercuric Chloride, Microchemical Reaction of Caffeine, ——, Theophylline, and their Derivatives with, 217
Theophylline, Mercuric Chloride, Microchemical Reaction of Caffeine, Theobromine, ——, and their Derivatives with, 217
Thermal Springs, Palestine, Analyses of Waters from, in, 102
———— Value, Turpentine Oil, Bromine, of, 314
Thermo-Electric Method, Platinum Ware, for the Determination of the Purity of, 381
Thermometer Bulb Method, Determination of Melting-Points (of Fats, etc.) by the, 61
———— Corrections, Boiling-Points without, Apparatus for Determining, 420
————, Vacuum-Jacketed, 154
Thermometers, Floating Equilibrium, Calibrating, by Means of, 153
Thermometry, Fixed Point in, Freezing-Point of Benzene as a, 510
Thermo-Regulator, Constant Temperature Chambers, for Electrically Regulated, 336
————, Improved, 104
————, Simple, 63
————, Temperatures Very Close to Room Temperature, for Thermostats at, 568
————, Vapour-Pressure, Electrical Contact, 154
Thermostat, High Temperature, 153
————, Smoke and Fume Monitors, Precipitators, and Recorders, Instantaneous, and, 567
Thermostats, Electrolytic Heating and Regulation of, Method for, 64
————, Thermo-Regulator for, at Temperatures Very Close to Room Temperature, 568
Thiocyanates, Bromides in Presence of, and Ferrocyanides, Detection of, Action of Chromic Acid on Cyanogen Bromide, 412
————, Bromides in Presence of, and Ferrocyanides, Detection of, 187
————, Ferro- and Ferricyanides and, Detection of Cyanides in Presence of, 365
Thiosulphate, Free Sulphuric Acid in Presence of, or Sulphuric Acid, Estimation of, 279
————, Sulphite, Estimation of, in Presence of, 101
Thiosulphates, Sulphites, Detection and Approximate Estimation of Traces of, in, 147
"Thomas" Meal, Error in the Analysis of, Sources of, 377
Thorium, Monazite Sand, Estimation of, in, 378
Timbers, Well-Preserved, Quantity and Character of Creosote in, 496
Tin, Copper and, Groups, Separation of the, 371
——, Copper, and Silicon Alloys, Analysis of, 500

Tinctures, Alcohol in, Rapid and Simple Method for the Estimation of, 393
Titanium, and Chromium, Volumetric Estimation by Means of a Modified Reductor of, 419
——— Chloride, Nitro-Compounds and Nitroso-Compounds, Volumetric Estimation of, by Means of, 134
———, Ferric Chloride, Volumetric Estimation of, by Means of, 563
———, Iron, Use of Ammonium Salt of Nitrosophenylhydroxylamine ("Cupferron") in Quantitative Separation of, from, 332
———, Phosphate, Estimation of, as, 279
———, Tobacco Ash, and Rare Metals in, 43
———, Volumetric Estimation of, 102
Titration, Carbonic Acid, of Small Quantities of, 143
———, Weight of a Substance, Estimation by, of the, 194
Titrations, Iodimetric and Acidimetric, Picric Acid as a Standardising Substance for, 267
Tobacco, Italian, Barium and Arsenic in, 43
——— Resins, 220
———, Titanium and Rare Metals in, Ash, 43
Tobaccos, East Africa Protectorate, from the, 125
o-Tolidine, Free Chlorine, Effect of Ferric Salts and Nitrites on the, and Starch Iodide Tests for, 454
Toluene, "Tricresol" as a Substitute for, in Enzyme Work, 263
Trade Names, Synthetic Drugs, New Remedies, Photographic Developers, etc., Index List of, of, 533
Trichloroacetic Acid, Organic Compounds, Colour Reactions with, of, 410
"Tricresol," Toluene in Enzyme Work, as a Substitute for, 263
Tropics, Alcohol Estimation in the, 480
Tryptophane, Estimation of, 47
Tungsten, Ammonium Molybdate Assay for Lead, Effect of, on, 192
———, High-Grade Metallic, Analysis of, 505
——— Lamp, Concentrated Filament, Uses in the Laboratory of the, 194
——— Salts, Tanning Materials, Application of, to the Analysis of, 507
Tungstic Acid, Separation and Estimation of, 379
Turkey-Red Oil, Analysis of, 48
Twig and Leaf Oils, Cuban and Long-Leaf Pines, of, and Cone Oil of Long-Leaf Pine, 498

Unsaponifiable Constituents, Mowrah Fat, of, and the Detection of this Fat in Animal and Vegetable Fats, 310
———————, Oils and Fats and Lanolin, Estimation by Means of the Digitonin Method of the, of, 310
———————, Shea Butter, of, and their Relation to those of Mowrah Fat, 440
Uranium, Beryllium, Separation from, of, 325
——— Salts, Morphine and Phenols, Detection by Means of, of, 438
Urea, Blood, New Method for the Estimation of, in, 39
Estimation of, 412
————, 95
-, Manometric Method for the, 553
Identification of, and its Precipitation from Very Dilute Solutions, 48
Quantitative Gravimetric Estimation of, 268
Urine, Gravimetric Estimation of, in, 362
Urease, Castor Bean, Experiments with. (Studies on Enzyme Action. XI.), 553
Urine, Allantoin in, Estimation of, 263

Urine, Characteristic Odour of, " Urinod," the Cause of the, 554
——, Different Sulphur Compounds, Rapid Estimation in, of, 101
——, Ethereal and Inorganic Sulphates, Volumetric Method for the Estimation in, of, 319
——, Free and Combined Amino-Nitrogen in, Gas-Volumetric Estimation of, 175
——, Guaiacol, Detection in, of, 360
——, Indigo-Forming Substances in, 39
——, Lead, Estimation of Small Quantities of, particularly in, in Cases of Lead Poisoning, 495
——, Mercury, Electrolytic Method for Estimation in, of, 225
——, Metals Present in, Influence of, on the Velocity of the Reduction Tests for Dextrose, 131
——, Oxyproteic Acid Fraction, Quantitative Estimation in, of the, 361
——, *p*-Cresol Tyrosinase Reaction, Detection of Peptides in, by the, 555
——, Picric Acid in, Detection of, 224
——, Proteins in, Centrifugal Method for Estimating, 40
——, Sugar in, Simple Colorimetric Method for Estimation of, 224
——, Sugars in, in Cases of Pentosuria, 40
——, Urea, Gravimetric Estimation in, of, 362
——, Various Preservatives for, Comparison of, 177
——, Violet Sodium-Nitro-Prusside Reaction of, 264
——, Volatile Substances of, 554
" Urinod," Urine, the Cause of the Characteristic Odour of, 554

" Vacuum Distillation " Method, Formic Acid, for the Detection of, 321
Vacuum-Jacketed Thermometer, 154
d-Valine, Separation of *d*-Alanine and, 178
Vanadium, Volumetric Estimation of, 148
(Vanilla Extracts: Acetanilide, Saccharin, and Caffeine, Analysis of, and Extraction of.) Study of Methods of Extraction by Means of Immiscible Solvents from Point of View of Distribution Coefficients, 354
Vanillin, Determination of, 220
——, Reactions of, 315
Vapour-Pressure Thermoregulator, Electrical Contact, 154
Vapour Pressures, Dynamical Methods, New Method for the Determination of, and an Examination of a Source of Error in Certain, 511
——, Measuring Partial, of Liquid Mixtures, Rapid Laboratory Method of, 569
Vapours, Bromine in Gases and, Detection of. VII.: Communication, 271
——, Paint, Composition of, 265
Varnish, Body and Solvent in, Quantitative Determination of, 560
Varnishes, Rosin, Estimation in, Oils, and Soaps of, 228
Vegetable Fats in Animal Fats, Detection of, 32
—— Fats, Unsaponifiable Constituents of Mowrah Fat and the Detection of this Fat in Animal and, 310
Oils, Diazonitro Reaction of, 228
—— Yeast, and Meat Extracts, Distribution of Nitrogen in, 444
Vegetation, Sulphurous and Sulphuric Acids in the Atmosphere, On Damage Caused to, by, 203
Verband Deutscher Elektrotechniker, Loading in Caoutchouc Mixtures, Estimation of, According to the Standards of the, 91
Vessels, Nitric Acid, Resistance of Platinum, to Hot, 382
Viking Period, Beeswax of the, 40

Vinegar, Cider, Furfural in, 256
Vinegars, Cider, Made by Generator Process, Composition of, 36
Vinometer, Bernadot's, 457
Violet, Sodium-Nitro-Prusside Reaction of Urine, 264
Viscometer, General Scientific and Technical Purposes, New, for, 284
Viscosimeter, New, 155
Viscosity, Determination of, 104
———, Milk, Determination of the, of, as a Means of Detecting the Addition of Water, 400
———, Mineral Oil Mixtures, Method for Calculating, of, 446
———, Oils, of, 185
———, Refined Syrups and Molasses, Determination of the, of, 220
———, Rubber Solutions, of, 322
———, Tars, Relation between the Melting-Point and, of Refined, 369
Volatile Liquids, Combustion of Very, Apparatus for the, 567
——— Oil of *Calicanthus Floridus*, 558
———, Hops, Aroma of. Study of the, with Relation to the Geographical Sources of the Hops, 355
——— Oils, Aromatic Plants, Drugs, and Spices, Estimation of, in, by the Tailameter, 35
——— Substances of Urine, 554
Volatility, Oils, Apparatus for Testing the, or Oxidisability of, 334

Ware, Purity of Platinum, Thermo-Electric Method for the Determination of the, 381
Waste Acids, Nitrocellulose Manufacture, Analysis of, from, 183
Water-Alcohol Solutions, Acids in, Inversion of Cane Sugar by, 323
———Analysis, Interferometer in, the Use of the, and Estimation of Colloids in Effluents, 325
———, Atmospheric Oxygen, Solubility in, of, 55
——— Board, Metropolitan. Tenth Research Report, 514
—. Eleventh Annual Report for the Year Ended March 31, 1914, 512
—, Cheese, Estimation of, in by Mai and Rheinberger's Method, 171
—, Crude Petroleum, Oil-Fuel, and Similar Substances, Two Rapid Methods of Estimating, in, 529
—, Dorton Spa, The, of, 210
—, Dry Starch, Soluble in Cold, Preparation of, for Use as an Indicator, 451
—, Examination, Fermentation Test at 45° C. as an Aid in, 129
—, Foods and Feeding-Stuffs, Rapid Estimation of, in, 36
——Gas Tar, Composition of, 369
—, Hardness in, Estimation of, 379
—, Hydrogen Sulphide in, New Apparatus for Estimation of. Part I., 457
—, Iron, Colorimetric Estimation in, of, 333
—, Manganese, Colorimetric Estimation in, of Small Quantities of, 54
—, Manganese in, Colorimetric Estimation of Traces of, 380
—, Milk, Determination of the Viscosity of, as a Means of Detecting the Addition of, 400
—, ——, The Detection of Added, in, 432
—, Mineral Springs, Detection and Estimation of Gaseous Hydrocarbons in the, of, 227
—, Oxygen in, Estimation of Dissolved, 55
—, ———, Field Method for Determining Dissolved, 234
—, Solid and Liquid Fuels, Estimation of, in, by Distillation with Xylene, 141

Water, Steel Subjected to Alternate Exposure to Air and Fresh, Tests of Paints for, 54
——, Sulphates, Estimation of, of Crystallisation in, 193
—— Supplies, Results of the Hypochlorite Disinfection of. A Comparison of the Reduction of the Different Members of the *Bacillus Coli* Group, 443
Waters, Aerated, Detection of Saponin in, 440
——, Free Carbon Dioxide in Mineral, Apparatus for the Approximate Estimation of, 238
——, Mineral, Fluorine in, 401
——, Mineral, Formation of Sediment in Bottled. Estimation of Silica, Iron, and Aluminium in Mineral, 126
——, Natural, Applicability of Conductivity Methods in Examination of, 149
——, Spring, Fontactoscope for Measuring the Radio-Activity of, 423
——, Thermal Springs in Palestine, Analyses of, from, 102
Wax, Petroleum Oil and Paraffin, Analysis of, 43, 266
Waxes, Flax and Hemp, from, 560
——, Montanin and Montana, 93
Weather, Nitric Acid and of Nitrous Acid in the Rainfall, Influence of, Conditions upon the Amounts of, 501
Weight, Titration, Estimation of the, of a Substance by, 194
Wet Strengths, Paper and Paper Yarns, Dry and, of, 322
—— Way, Carbon, Estimation by the, of. Simultaneous Estimation of Halogens, 132
——, Carbon in Aliphatic Substances, Volumetric Estimation of, in the, 556
Wheat Flour, Detection of Small Quantities of Rye Flour in, 400
——, ——, Estimation of the Proteins in, 550
——, ——, the Proteins Soluble in Salt Solutions in, Quantitative Estimation of, 262
——, Milling of, and Testing of Flour, 358
——, Proximate Analysis of, 441
Whistle, Fire-Damp, 193
White Fir. Oils of the *Coniferæ*, Leaf and Twig, and Bark Oils of, 558
—— Metals, Electro-Analysis of, New Method for, 52
Wine Analyses, Chemico-Physical Principles, Method of Expressing Results of, according to the Latest, 127
——, Extract in, Estimating, 221
——, Glycerol, Estimation in, of, 87
——, Hexamethylenetetramine, Detection in, or Milk of, 125
——, Lactic Acid, Estimation in, of, 37
——, —— in, Estimation by Moslinger's Barium Chloride Method of, 397
——, Malt, 128
——, Proportion of Acid and the Degree of Acidity of, Difference between the, 221
——, Sulphurous Acid, Estimation in, of Total, 87
Wines, Coal-Tar Dyes, Detection in, of, 358
——, Concentrated Musts and, Organic Phosphoric Acid Compounds in, 398
Wood Preservatives (Creosote Oil), Report of the Committee on, 497
——, Pulp, Chemical Valuation of, for, 451
——, Treated, Method for the Estimation of Zinc in, 564
Woods, Pine, Fractional Distillation of Mixed, 48
Worts, Specific Gravities of Malt, and Beers, Temperature Corrections to be Applied to the, 256

Xylene, Water in Solid and Liquid Fuels, Estimation of, by Distillation with, 141
Xylose, Pentoses, and Arabinose, Methods of Estimation of Carbohydrates. III.: The Cupric-Reducing Power of the, 555

Yarns, Paper and Paper, Dry and Wet Strengths of, 322
Yeast, Glycogen, Determination in, of, 129
——, Vegetable, and Meat Extracts, Distribution of Nitrogen in, 444
Yeasts, Top and Bottom, Method of Preparing Strong Invertase Solutions from, 443
Yellow Mercuric Oxide, Estimation of Carbon Monoxide by Means of, 226
Yttrium Earths, Separation of Yttrium from the: Part I., 280; Part II., 420

Zeiss Interferometer, Gases, for the Analysis of, 103
Zelmanowitz's Apparatus, Liquids, Modification of, for Extracting, 64
Zinc, Coinage Bronze, Estimation of, in, by Volatilisation, 236
——, Determination, Silver-Plated Copper-Gauze Electrode in, 382
——, Iron, Aluminium, Chromium, and Manganese, Investigation of the Methods of Analysis and Separation of, 272
——, Treated Wood, Method for the Estimation of, in, 564
Zirconium, Iron and Aluminium, Separation of, from, with the Aid of the Ammonium Salt of Nitrosophenylhydroxylamine ("Cupferron"), 565

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THE ANALYST.

OBITUARY.

JAMES CARTER BELL, A.R.S.M.

On July 1 there passed away, at his residence—The Cliff, Higher Broughton, Manchester—James Carter Bell, a man who, for close upon half a century, has quietly and unobtrusively given of his best in the public service and to private enterprise.

The late Mr. Carter Bell was born at Hastings in 1839, and was educated privately in the South of England. He studied chemistry at the Royal School of Mines under A. W. Hofmann, and during the five years over which his academic studies were spread, he enjoyed the unique opportunity of sitting under such stalwarts as Huxley, John Percy, Warington Smyth, and Tyndall. After taking his degree of Associate of the Royal School of Mines, he had thoughts of proceeding with the purely academic side of Chemistry, and gained some experience as a Lecturer at the old Regent Street Polytechnic; but the practical application of the science appealed ultimately to his enthusiasm and activities, and he consequently accepted an offer in 1865 to act as chemist to Messrs. Peter Spence and Sons, the proprietors of the Manchester Alum Works. With this firm he did sterling work, and retained the position for many years, first as resident, and later as consulting chemist. In the interests of a gradually increasing private practice, he opened a laboratory in the centre of Manchester, and early in 1876 was appointed Public Analyst for Salford, and in October of the same year County Analyst for Cheshire. Similar appointments under the Glossop, Stalybridge and Birkenhead Corporations followed in due course.

Mr. Carter Bell was one of the founders of the Society of Chemical Industry and was an active member of the Society of Public Analysts in its earliest days, and also of the Institute of Chemistry. But of all Societies, none claimed more of his time and attention than the Society of Chemical Industry. The Manchester Section of that Society has been fortunate from its inception in having the sympathy and practical support of men eminent in pure and applied chemistry, but for faithful services most cheerfully rendered, and onerous duties unobtrusively carried out, the name of Carter Bell will ever be held in grateful remembrance by all who have watched with pride the gradual growth and increased sphere of usefulness of this Section. For close upon a quarter of a century, he acted as its local Secretary, giving freely of his time, rousing lethargic members to activity by his example, and developing the social side of the Society on all occasions. It was but fitting that, on his retirement from his secretarial duties, he should occupy the position of Chairman, which he held in 1904 and 1905. During these many years he took an active part on the Publication Committee of the Society, and devoted himself most assiduously to maintaining the highest traditions of the office. With the unfortunate indisposition from which Mr. Carter Bell suffered during the past seven years, he necessarily took a less active part in the work of the Society, but his interest never flagged, and to the end he closely followed its progress as recorded in the pages of its Journal.

Mr. Carter Bell was also one of the founders of the Manchester Chemical Club, being in turn Honorary Secretary, Treasurer, and President. Amongst other professional duties which devolved upon him were those of Consulting Analyst to the Silk Association and Advising Chemist to the Rivers Committee of the Salford Corporation. In the early days when the question of sewage disposal had scarcely been touched upon, Mr. Carter Bell carried out elaborate tests of the many precipitation and filtration methods of sewage purification at the Corporation Works at Weaste, and, having a wide experience in the practical problem involved, he could see farther than the majority where processes could be modified and simplified in order to arrive at the best results. Up to the last he guided the Committee in its frequent deliberations, ever anxious to safeguard its welfare in every direction.

As he acted as Public Analyst for so many authorities, it would appear scarcely necessary to enlarge upon his services in the interests of public health, but it is essentially in this sphere that lies his greatest claim to recognition. The keynote of his life was thoroughness. His attention to detail amounted almost to a passion, [illegible] the highest ideals of his office, he would with judicial care marshal his facts and act upon them without fear or favour. No greater tribute can be paid to his accuracy of work than that in the many cases of samples of food and drugs disputed and referred to the Somerset House Authorities, his official certificates were without exception upheld. He was very much alive to the ever-increasing refinements in adulteration, and his reports to the Cheshire County Council show the methods adopted in coping with them.

The following is a list of Mr. Carter Bell's principal contributions:

1878. "Note on the Detection of Alum in Flour"; "Notes on Milk Analysis." 1879. "Analysis of Bread Flour"; "Differences in the Quality of Milk between the End and the Beginning of Delivery"; "Test for Morphia." 1880. "The Composition of Unfermented Wines"; "The Manufacture of Citric Acid." 1881. "Further Notes on Milk"; "Analysis of Grape Juice." 1882. "The Composition of the Mineral Matter of Tea"; "Manufacture of Chloride of Sulphur"; "Action of Sulphuretted Hydrogen on Compounds containing Oxide of Iron." 1883. "Examination of Beer from Beerhouses and Brewers." 1898. "On the Oxygen Test for Sewage and Effluents as carried out in the Laboratory of the Mersey and Irwell Joint Committee" (*J. Soc. Chem. Ind.*, pp. 11 and 425); "On the Comparative Effect of Lime and other Chemicals upon Sewage" (*ibid.*, p. 545). 1903. Municipal Chemistry" (*ibid.*, p. 1077).

Apart from his work, the personality of the man won for him the sincere respect of all with whom he came in contact. He was of a courteous and kindly disposition, and there are many who will feel keenly the loss of a sympathetic friend and adviser. The martial spirit had no place in Mr. Carter Bell's character; rather was he of a quiet disposition, and occasionally somewhat restrained in manner, but at all times there was the reverent regard for all things beautiful. Year after year he would take his hard-earned holiday tramping through the highways and byways of his beloved France, profiting by the secrets never withheld from the true Nature worshipper, and returning to his duties with renewed enthusiasm and activity. He leaves a widow, son, and daughter to mourn his loss, and the greatest sympathy will be extended to them for the loss of "one who had done his work and held his peace and had no fear to die."

E. Knecht.

* * * * *

INTERNATIONAL ATOMIC WEIGHTS, 1914.

		O=16.			O=16.
Aluminium	Al	27·1	Molybdenum	Mo	96·0
Antimony	Sb	120·2	Neodymium	Nd	144·3
Argon	A	39·88	Neon	Ne	20·2
Arsenic	As	74·96	Nickel	Ni	58·68
Barium	Ba	137·37	Niton (radium emanation)	Nt	222·4
Bismuth	Bi	208·0	Nitrogen	N	14·01
Boron	B	11·0	Osmium	Os	190·9
Bromine	Br	79·92	Oxygen	O	16·00
Cadmium	Cd	112·40	Palladium	Pd	106·7
Cæsium	Cs	132·81	Phosphorus	P	31·04
Calcium	Ca	40·07	Platinum	Pt	195·2
Carbon	C	12·00	Potassium	K	39·10
Cerium	Ce	140·25	Praseodymium	Pr	140·6
Chlorine	Cl	35·46	Radium	Ra	226·4
Chromium	Cr	52·0	Rhodium	Rh	102·9
Cobalt	Co	58·97	Rubidium	Rb	85·45
Columbium	Cb	93·5	Ruthenium	Ru	101·7
Copper	Cu	63·57	Samarium	Sa	150·4
Dysprosium	Dy	162·5	Scandium	Sc	44·1
Erbium	Er	167·7	Selenium	Se	79·2
Europium	Eu	152·0	Silicon	Si	28·3
Fluorine	F	19·0	Silver	Ag	107·88
Gadolinium	Gd	157·3	Sodium	Na	23·00
Gallium	Ga	69·9	Strontium	Sr	87·63
Germanium	Ge	72·5	Sulphur	S	32·07
Glucinum	Gl	9·1	Tantalum	Ta	181·5
Gold	Au	197·2	Tellurium	Te	127·5
Helium	He	3·99	Terbium	Tb	159·2
Holmium	Ho	163·5	Thallium	Tl	204·0
Hydrogen	H	1·008	Thorium	Th	232·4
Indium	In	114·8	Thulium	Tm	168·5
Iodine	I	126·92	Tin	Sn	119·0
Iridium	Ir	193·1	Titanium	Ti	48·1
Iron	Fe	55·84	Tungsten	W	184·0
Krypton	Kr	82·92	Uranium	U	238·5
Lanthanum	La	139·0	Vanadium	V	51·0
Lead	Pb	207·10	Xenon	Xe	130·2
Lithium	Li	6·94	Ytterbium (Neoytterbium)	Yb	172·0
Lutecium	Lu	174·0	Yttrium	Yt	89·0
Magnesium	Mg	24·32	Zinc	Zn	65·37
Manganese	Mn	54·93	Zirconium	Zr	90·6
Mercury	Hg	200·6			

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

An ordinary meeting of the Society was held on Wednesday evening, December 3, in the Chemical Society's Rooms, Burlington House.

The President, Mr. L. Archbutt, F.I.C., occupied the chair.

The minutes of the previous ordinary meeting were read and confirmed.

Certificates of proposal for election to membership in favour of Messrs. A. M. Bailey, A.I.C., A. L. Barton, T. S. Haines, A.I.C., and A. G. A. Miller, B.Sc., were read for the second time; and certificates in favour of Messrs. Rowland Holliday Ellis, A.I.C., "Lynwood," Thorpe Road, Selby, Analyst to the Olympia Oil and Cake Company, Ltd., Selby; and Armand de Waele, "Rosemead," Sidney Road, Staines, Chemist to the Linoleum Manufacturing Company, Ltd., were read for the first time.

Messrs. J. J. Eastick, A.R.S.M., F.I.C., and J. F. Millar, were elected members of the Society.

The following papers were read: "Sulphuretted Hydrogen from Artificial Graphite," by W. H. Woodcock and Bertram Blount, F.I.C.; "The Rate of Liberation of Hydrocyanic Acid from Linseed," by S. Hoare Collins, M.Sc., F.I.C., and H. Blair; "The Determination of Sulphur in Rubber," by Henry P. Stevens, M.A., Ph.D., F.I.C.; "The Composition of Palm Kernel Oil," by George Davidson Elsdon, B.Sc.; "Notes on the Insoluble Bromide Test for Drying Oils," by J. A. L. Sutcliffe; and "The Determination of Strychnine in the Presence of Quinine," by Charles Simmonds, B.Sc.

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THE PROPERTIES OF SOME CHLORHYDROCARBONS AND THEIR USES IN CHEMICAL ANALYSIS.

By L. GOWING-SCOPES.

Part I.

Investigation carried out under the Society's Analytical Investigation Scheme.

(*Read at the Meeting, November* 5, 1913.)

The literature on the chlorethanes and the chlorethylenes is very extensive. Up to about four or five years ago these substances were comparatively rare; but in 1909 G. Koller communicated a paper to the Seventh International Congress of Applied Chemistry calling attention to the fact that the chlorethanes and chlorethylenes were being manufactured in this country, and were being used on a large scale commercially. Since then they have been studied by V. H. Veley (*Proc. Roy.*

Soc., 1910, B. 82, 217-225) as to their anæsthetic and physical properties; by the author in a paper dealing with the uses of trichlorethylene in analytical chemistry (ANALYST, 1910, 238-245), and by several others who will be mentioned under the various subheadings to which the subjects of their papers particularly apply.

Physical Properties.—The physical properties of the chlorhydrocarbons have been studied by Herz and Rathmann (*Chem. Zeit.*, 1912, **36**, 1417; 1913, **37**, 621; *Zeitsch. Electrochem.*, 1913, **19**, 589) and V. H. Veley (*loc. cit.*).

The table given is compiled from various sources; the figures for chloroform and carbon tetrachloride are given for reference.

Extended figures for densities, etc., are given in the above-mentioned papers by V. H. Veley and the author (*loc. cit.*) respectively.

Some of the boiling-points recorded are still a little uncertain, but the values are probably good approximations:

	Boiling-Point. °C.	Freezing-Point. °C.	Density 25° C.	Coefficient of Expansion.
Chloroform	61·5	—	1·4791	0·001257
Carbon tetrachloride	76·7	—	1·5835	0·001227
Dichlorethylene Cis.	48·8 (at 763·0 mm.)	—	1·3328	0·001360
,, Trans.	59·8 (at 763·0 mm.)	—	1·3545	0·001270
Trichlorethylene	87·5	- 73	1·4542	0·001193
Tetrachlorethylene	121·0	- 19	1·6080	0·001078
Tetrachlorethane	147·2	- 36	1·5881	0·000998
Pentachlorethane...	159·1	- 22	1·6712	0·000909
Hexachlorethane	185·5 (at 776·7 mm.)	- 187	2·01 (?)	—

	Heat of Vaporisation (Calories).	Refractive Index.	Viscosity.			Specific Heat.
			25° C.	50° C.	70° C.	
Chloroform	—	1·449 (15° C.)	—	—	—	—
Carbon tetrachloride	—	1·464 (15° C.)	—	—	—	—
Dichlorethylene Cis.	6930	—	0·437	—	—	—
,, Trans.	7268	—	0·510	0·671	—	—
Trichlorethylene ...	7436	1·47914 (17° C.)	0·615	0·812	0·976	0·223
Tetrachlorethylene ...	8554	—	0·940	1·194	1·404	0·216
Tetrachlorethane ...	9134	1·49559 (17° C.)	1·808	2·033	2·154	0·268
Pentachlorethane ...	8829	—	2·432	2·695	2·789	0·266
Hexachlorethane ...	—	—	—	—	—	—

Purification.—The only satisfactory method of removing chlorine and hydrochloric acid is to shake vigorously with a strong solution of ammoniacal silver nitrate. This is to neutralise the acid formed during the reaction, and the trichloracetyl chloride present as an impurity. The two liquids are then separated as completely as possible, washed free from ammonia, and again separated. The chlorhydrocarbon is allowed to stand over good quicklime for about twelve hours to remove excess of water, and then distilled. The distillate is again fractionated from phosphorous pentoxide. As these compounds decompose in the presence of light, moisture, and air, the anhydrous distillate should be collected in small bottles, filled to the stopper, and stored in the dark. A small quantity of anhydrous potassium carbonate may be added to keep down the acidity, but with tetrachlorethylene, which it decomposes, this must not be used.

Distillation Experiments.—Experiments are in progress to ascertain what binary and ternary mixtures are formed with water, alcohols, acids, etc., with a view to effecting useful separations. Up to the present it has been indicated that water forms with dichlorethylene a binary mixture, boiling about 49° C. (uncor.), and with trichlorethylene, a mixture boiling about 78·5° C. (uncor.).

G. Chavanne (*Bull. Soc. Chem. Belg.*, 1913, **27**, 205-209; ANALYST, 1913, **38**, 424-425) has found that the two isomeric dichlorethylenes form binary mixtures with absolute alcohol, and ternary mixtures with water and alcohol, boiling at low temperatures. They can be used instead of benzene in Young's method of obtaining absolute alcohol. The following table gives the composition of the mixtures:

	Isomer of Boiling-Point, 48·35° C.				Isomer of Boiling-Point, 60·25° C.			
	Boiling-Point.	Composition.			Boiling-Point.	Composition.		
	°C.	$C_2H_2Cl_2$.	Alcohol.	Water.	°C.	$C_2H_2Cl_2$.	Alcohol.	Water.
Ternary mixture ...	44·4	94·5	4·4	1·1	53·8	90·50	6·65	2·85
Binary mixture ...	46·5	94·0	6·0	—	57·7	90·20	9·80	—
Binary mixture ...	45·3	98·1	—	1·9	55·3	96·65	—	3·35

Action of the Chlorhydrocarbons on some Common Metals.—Loose pieces of metal were placed in the bottom of the flask, which was fitted with a condenser, the metal being completely covered with the liquid. Another piece was suspended in the neck of the flask so as to be subjected to the vapours only. The contents of the flask were boiled for a day, and the metal examined as to whether it had been attacked. If it had not been attacked, it was replaced, some water was added, and the process repeated, giving the combined action of steam and vapour. The following were the results obtained:

Metal.	$C_2H_2Cl_4$.	C_2HCl_5.	$C_2H_2Cl_2$.	C_2HCl_3.	C_2Cl_4.
Sodium ...	Very vigorous reaction.	Hardly attacked.	Slow reaction.	Slow reaction.	Fairly vigorous reaction.
Copper ...	Attacked dry.	Attacked dry.	No action; slightly attacked in presence of water.	No action; slightly attacked in presence of water.	Slightly attacked; coating of chloride in presence of water.
Silver ...	Coated with chloride.	Coated with chloride.	Slightly coated with chloride.	No action; slightly attacked.	Coated with chloride.
Magnesium	Metal dulled.	Metal dulled.	No action; slightly attacked in presence of water.	No action; slightly attacked in presence of water.	No action; slightly attacked in presence of water.
Zinc ...	No action attacked with water.	Attacked.	No action; slightly attacked in presence of water.	No action; slightly attacked in presence of water.	Slight action.
Mercury ...	Forms Hg_2Cl_2.	Forms Hg_2Cl_2.	No action; Hg_2Cl_2 in presence of water.	Forms Hg_2Cl_2.	Forms Hg_2Cl_2.
Aluminium	Vigorous reaction.	Very violent reaction.	No reaction; no action in presence of water.	No action; slightly attacked in presence of water.	No action; slightly attacked in presence of water.
Tin	Metal dulled.	Attacked.	No action; no action in presence of water.	Slight action.	Slight action.
Lead ...	Quickly coated with chloride.	Quickly coated with chloride.	No action; quickly coated with chloride in presence of water.	Quickly coated with chloride.	Quickly coated with chloride.
Antimony ...	Slightly etched.	Slightly etched.	No action; faintly attacked in presence of water.	Faint action; attacked in presence of water.	Slightly etched.
Iron ...	No action; attacked in presence of water.	No action; attacked in presence of water.	No action; attacked in presence of water.	No action; attacked in presence of water.	Attacked; attacked in presence of water.
Nickel ...	No action; attacked in presence of water.	Attacked.	No action; no action in presence of water.	No action; slightly attacked in presence of water.	No action; slightly attacked in presence of water.

The action of aluminium on the chlorethanes is very interesting, and is being investigated further. It is suggested as an explanation that aluminium trichloride is first formed in small quantities, which in turn reacts catalytically with the chlorethanes, causing the remainder of the liquid and the metal to react, a final double compound of aluminium trichloride and the chlorethanes being formed. It is hoped that this reaction may be utilised to remove the chlorethylenes from the chlorethanes. Experiments are also in progress to see whether hexachlorethane also reacts in this manner, as it would be extremely useful for removing this substance from trichlorethylene when used for fat estimations, etc.

The action of sodium on trichlorethylene is important, a small quantity of a spontaneously inflammable gas being liberated, which suddenly explodes in the condenser tube, leaving a copious deposit of carbon. Possibly this gas may be acetylene chloride, C_2HCl.

Action of some other Inorganic Substances on the Chlorhydrocarbons.—Red and yellow phosphorus are practically insoluble, although at the boiling-point a little of the yellow variety dissolves in some of them and crystallises out on cooling. Sulphur is found to be soluble in tetrachlorethane and pentachlorethane, large quantities dissolving on heating and crystallise out on cooling. In the chlorethylenes, "flowers of sulphur" are not so soluble, particularly in tetrachlorethylene, which only dissolves very small quantities. If, however, the sulphur be melted, tetrachlorethylene dissolves large quantities, which crystallise on cooling.

Hydrochloric acid is only very slightly soluble in the chlorhydrocarbons. Hot sulphuric acid attacks them only very slowly, and does not mix with them. Nitric acid is only slightly soluble, and does not appear to react with any of them. Nitrous acid is soluble in all, forming a green solution; in some cases a slow reaction appears to take place. Phosphorous pentoxide turns brown with dichlorethylene and tetrachlorethane, but has very little action on the others. Ferric chloride is slightly soluble. Aluminium trichloride reacts vigorously, in some cases apparently forming a new compound (*loc. cit.*). Hydroxylamine hydrochloride is insoluble. Ammonia dissolves fairly readily. Water is soluble only in small quantities; the exact proportion is being determined.

The Solubility of Organic Compounds in the Chlorhydrocarbons.—The solubility of 248 organic substances was determined. The results are set forth in the tables on pp. 9-14. They have been made roughly quantitative by using the following symbols to indicate the degree of solubility:

VSS: Very slightly soluble (0·1 per cent.). SS: Slightly soluble (up to 1 per cent.). S: Soluble (up to 7 per cent.). VS: Very soluble (up to 20 per cent.). ES: Extremely soluble (above 20 per cent.). M: Miscible in all proportions. I: Insoluble.

The solubilities in all cases refer to solution in the cold; many substances dissolve very readily on boiling, but the substance separates out completely on cooling.

	Tetrachlor-ethane.	Pentachlor-ethane.	Dichlor-ethylene.	Trichlor-ethylene.	Tetrachlor-ethylene.
HYDROCARBONS :					
Petroleum Spirit, Benzoline, and Kerosene	M	M	M	M	M
Vaseline	S	S	S	S	S
Paraffin Wax	VS	VS	VS	VS	VS
Benzene, Toluene, and Xylene	M	M	M	M	M
Naphthalene	VS	VS	ES	VS	VS
Anthracene	SS	SS	S	S	SS
Phenanthrene	S	S	VS	S	SS
Acenaphthene	ES	ES	ES	ES	ES
Diphenyl	ES	ES	ES	ES	ES
Turpentine	M	M	M	M	M
Naphthalene Picrate ...	SS	VSS	VS	VSS	VSS
ETHERS :					
Ethyl ether and Guaiacol	M	M	M	M	M
β-Naphthylmethyl ...	S	S	S	S	S
Eugenol	M	M	M	M	M
ALCOHOLS :					
Methyl, Ethyl, Propyl, Butyl, and Amyl	M	M	M	M	M
Cetyl	SS	SS	S	S	S
Allyl	M	M	M	M	M
Glycerol	I	I	I	VSS	VSS
Mannitol	I	I	I	I	I
Benzyl	M	M	M	M	M
Borneol	S	S	S	S	S
Menthol	ES	VS	ES	ES	ES
Benzhydrol	ES	ES	ES	ES	ES
PHENOLS :					
Phenol	ES	ES	VS	VS	VS
Meta Cresol	M	M	M	M	M
α- and β-Naphthol and Thymol	S	S	S	S	S
Carvacrol	M	M	M	M	M
Catechol...	I	I	VSS	VSS	VSS
Resorcinol	I	I	SS	VSS	VSS
Hydroquinone	I	I	VSS	I	I
Pyrogallol	I	I	SS	SS	SS
Phloroglucinol	I	I	VSS	I	I
Saliginen	S	S	S	S	SS

	Tetrachlorethane.	Pentachlorethane.	Dichlorethylene.	Trichlorethylene.	Tetrachlorethylene.
ALDEHYDES:					
Formaldehyde	S	S	S	S	S
Paraformaldehyde ...	I	I	S	I	I
Acetaldehyde	M	M	M	M	M
Metaldehyde	I	I	S	S	I
Paraldehyde, Salicylaldehyde, Anisaldehyde, and Cinnamic Aldehyde	M	M	M	M	M
Vanillin	S	S	ES	S	SS
Furfurol	M	M	M	M	M
Chloral Hydrate ...	SS	VSS	S	S	S
Butyl Chloral	SS	I	S	S	I
Paradimethylamidobenzaldehyde	VS		ES	VS	VS
KETONES AND QUINONES:					
Acetone	M	M	M	M	M
Camphor...	S	—	S	S	—
Benzoquinone	I	I	VSS	VSS	I
Alizarin	SS	I	VSS	VSS	VSS
ACID ANHYDRIDES:					
Acetic	M	M	M	M	M
Phthalic	SS	SS	SS	SS	SS
Camphoric	S	S	S	S	S
ACIDS AND ESTERS:					
Formic	M	SS	SS	SS	SS
Acetic, Propionic, and Caproic	M	M	M	M	M
Lauric	S	VS	VS	VS	VS
Palmitic and Stearic ...	S	S	S	S	VS
Oleic	M	M	M	M	M
Lactic, Oxalic, Malonic, Succinic, Sebacic, Malic, Tartaric, Citric, and Mucic					
Benzoic	S	S	S	S	S
Cinnamic	SS	SS	SS	SS	SS
Phthalic	I	I	I	I	I
Salicylic	I	I	VSS	VSS	I
Mandelic...	I	I	VSS	VSS	I
Gallic	I	I	I	I	VSS
Meconic	I	I	I	I	I
Camphoric	SS	VSS	SS	SS	VSS
Sulphanilic	I	I	I	I	I
Amyl Formate, Amyl Acetate, and Triacetin	M	M	M	M	M

	Tetrachlorethane.	Pentachlorethane.	Dichlorethylene.	Trichlorethylene.	Tetrachlorethylene.
Spermaceti and Methyl Oxalate	SS	S	S	S	S
Succino-Succinic Ester...	S	S	S	S	S
Sodioacetoacetic Ester...	I	I	I	I	I
Methyl Salicylate ...	M	M	M	M	M
Triphenyl Phosphate ...	S	S	S	S	S
CARBOHYDRATES:					
Typical carbohydrates (eleven examined)	I	I	I	I	I
GLUCOSIDES:					
Amygdalin	VSS	VSS	VSS	I	I
Salicin	I	I	S	VSS	VSS
HALOGEN DERIVATIVES:					
Chloroform, Carbon Tetrachloride, Tetrachlorethane, and Pentachlorethane	M	M	M	M	M
Hexachlorethane ...	VS	VS	VS	VS	VS
Dichlorethylene, Trichlorethylene, Tetrachlorethylene, Chlorbenzene, Orthochlortoluene, Ethyl Bromide, and Acetylene Tetrabromide	M	M	M	M	M
Iodoform	S	S	S	S	S
Paraiodotoluene ...	ES	VS	ES	ES	ES
Benzoyl Chloride ...	M	M	M	M	M
Orthochlorbenzoic Acid	SS	SS	SS	SS	SS
AMIDES AND ALLIED COMPOUNDS:					
Acetamide	VSS	VSS	S	S	VSS
Oxamide	I	I	VSS	VSS	VSS
Benzamide	SS	VSS	SS	SS	VSS
Urea	VSS	VSS	SS	VSS	VSS
Thiourea	I	I	VSS	I	I
Asparagin	I	I	VSS	VSS	I
Semicarbazide Hydrochloride	I	I	VSS	SS	SS
NITRO COMPOUNDS:					
Nitrobenzene	M	M	M	M	M
Metadinitrobenzene ...	S	S	ES	S	VSS
Nitronaphthalene ...	VS	VS	ES	VS	VS

	Tetrachlorethane.	Pentachlorethane.	Dichlorethylene.	Trichlorethylene.	Tetrachlorethylene.
Orthonitraniline	VS	S	ES	S	SS
Metanitraniline ...	SS	VSS	SS	SS	SS
Paranitraniline ...	S	SS	SS	SS	VSS
Orthonitrophenol	VS	S	S	S	S
Paranitrophenol ...	SS	SS	S	SS	VSS
Picric Acid	VSS	VSS	VSS	VSS	VSS
Orthonitrobenzaldehyde	—	—	SS	S	
Metanitrobenzaldehyde	S	S	ES	S	S
Paranitrobenzaldehyde	S	S	SS	SS	VSS
Orthochlornitrobenzene	VS	ES	ES	ES	ES
Pyroxyline	I	I	I	I	I
PURINE DERIVATIVES:					
Uric Acid ...	VSS	I	VSS	I	I
Ammonium Urate	I	I	VSS	I	I
Theobromine	VSS	I	S	S	VSS
Caffeine	VS	S	S	S	S
AMINO-ACIDS AND ALLIED COMPOUNDS:					
Glycine			SS	SS	I
Hippuric Acid			SS	I	I
Creatine		I	I	VSS	VSS
AMINO COMPOUNDS:					
Hexamethylenetetramine	VSS	VSS	VSS	VSS	VSS
Aniline	M	M	M	M	M
Paratoluidine	ES	VS	ES	VS	VS
β-Naphthylamine ...	S	SS	S	S	SS
Paraphenylenediamine...	VS	S	S	S	I
Diphenylamine	ES	ES	ES	ES	VS
Benzidine	SS	SS	S	S	I
Orthotolidine	ES	S	S	S	VS
Parachloraniline ...	S	VS	VS	VS	S
Tribromaniline	VS	VS	VS	VS	VS
Antipyrin H.I.	VSS	SS	VSS	VSS	SS
Aniline Hydrochloride ...	I	I	VSS	I	I
Metaphenylenediamine Hydrochloride and Metol					I
Phenylhydrazine ...	M	M	M	M	M
AZO AND DIAZO COMPOUNDS:					
Azobenzene	VS	ES	ES	ES	ES
Azoxybenzene and Diazoaminobenzene	ES	ES	ES	ES	ES

	Tetrachlor-ethane.	Pentachlor-ethane.	Dichlor-ethylene.	Trichlor-ethylene.	Tetrachlor-ethylene.
OSAZONES AND HYDRAZONES:					
Glucosazone	SS	VSS	I	VSS	VSS
Mannosehydrazone ...	VSS	SS	I	I	I
PYRIDINES, ETC.					
Pyridine and Quinoline	M	M	M	M	M
Carbazole	VSS	I	SS	SS	SS
ACYL SUBS. AMINES.					
Acetanilide	S	S	S	S	VSS
Parabromacetanilide ...	S	S	S	S	SS
COMPOUNDS CONTAINING C, H, S, N, AND O:					
Carbon Disulphide ...	M	M	M	M	M
Saccharin	I	I	S	SS	I
Gelatin, Caseine, Albumin, and Hæmoglobin	I	I	I	I	I
GUMS AND RESINS:					
Gums Acacia and Tragacanth	I	I	I	I	I
Resin	VS	VS	S	S	SS
Copal, Soft	S	SS	I	S	S
Copal, Zanzibar ...	SS	VSS	SS	SS	VSS
Kauri	VS	SS	SS	SS	SS
Bitumen	VS	S	S	VS	VS
Shellac	SS	VSS	SS	SS	SS
Dammar	VS	S	VS	VS	VS
Sandarach	S	SS	SS	SS	SS
Benzoin, White ...	ES	S	SS	VSS	VSS
Benzoin, Dark	VSS	I	I	I	I
Mastic	VS	VS	VS	VS	VS
Dragon's Blood ...	SS	SS	SS	VSS	VSS
Canada Balsam ...	ES	VS	ES	ES	ES
Gamboge	SS	SS	S	S	SS
DYES:					
Black.					
Japan Black	VS	I	S	VSS	I
Jet Black (BT) ...	SS	I	S	VSS	VSS
Nigrosin (5 C) (Spirit sol.)	VS	VSS	S	S	S
Nigrosin (Water sol.) ...	I	I	I	I	I
Nigrosin (G)	VSS	I	S	I	I
Blue.					
Victoria Blue (B) ...	VS	I	ES	SS	S
Victoria Blue (BS) ...	ES	SS	ES	S	I
Patent Blue (A)	S	I	S	I	I

	Tetrachlorethane.	Pentachlorethane.	Dichlorethylene.	Trichlorethylene.	Tetrachlorethylene.
Aniline Blue (BB) ...	S	VSS	ES	SS	I
Aniline Blue (II B) ...	S	SS	SS	VSS	VSS
Methyl Blue	I	I	VSS	I	I
Methylene Blue ...	VSS	I	S	I	I
Soluble Blue (I N) ...	I	I	I	I	I
Neptune Blue	S	I	S	I	I
Nicholson's Blue ...	VSS	I	VSS	VSS	I
Brown.					
Bismarck Brown ..	VSS		SS	I	I
Chrysoidine	I		S	S	I
Vesuvine (OOOL) ..	I		SS	I	I
Vesuvine (BL)	I		VSS	I	I
Green.					
Leaf Green	ES	SS	ES	SS	SS
Malachite Green ...	S	I	ES	S	I
Diamond Green (G) ...	VS	I	S	SS	VSS
Diamond Green (B) ...	VS	VSS	S	S	VSS
Ethyl Green	VSS	I	S	SS	SS
Spirit Green (II) .	S	SS	S	SS	I
Red.					
Rubine (N)	SS	I	S	I	I
Rosaniline	I	I	S	S	I
Saffranine Scarlet (B) .	S	I	ES	VSS	I
Saffranine Scarlet (G) .	S	VSS	VSS	VSS	I
Spirit Scarlet (G) .	VSS	VSS	S	I	I
Induline Scarlet ...	VSS	I	SS	VSS	I
Rhodamine (B extra) .	ES	SS	ES	SS	SS
Phenolphthaleïn .	SS	VSS	SS	VSS	VSS
Eosin (Yellowish) .	I	I	VSS	I	I
Eosin (Bluish)	I	I	I	I	I
Sudan (III)	ES	VS	ES	VS	VS
Cotton Scarlet	I	I	I	I	I
Violet.					
Methyl Violet (B extra)	VS	S	ES	S	I
Methyl Violet (6 B) ...	ES	SS	ES	S	VSS
Indigo Carmine ...	I	I	I	I	I
Yellow.					
Metanil Yellow (extra)	I	I	S	S	I
Auramine (OO)	S	VSS	S	SS	I
Eucrysine (RR) ...	SS	I	S	SS	VSS
Methyl Orange	I	I	VSS	VSS	I
Fluorescein	VSS	VSS	I	I	I

The slight solubility of anthracene in some of the solvents will be noted, while phenanthrene is fairly soluble in all except tetrachlorethylene.

The solubility of naphthalene picrate in dichlorethylene suggests the possibility of separations of the hydrocarbons by means of the picrates. Further work is being done on this subject.

The monohydric alcohols are soluble in all the solvents, while the polyhydric alcohols are insoluble. The slight solubility of cetyl alcohol in the chlorethanes is interesting.

Monohydric phenols are soluble, while polyhydric phenols are practically insoluble; most of the polyhydric phenols tried are soluble in the hot solvent, but crystallise out completely on cooling. Saligenin is an exception in all the solvents except tetrachlorethylene. Experiments are being carried out to see whether the monohydric and polyhydric phenols may be separated quantitatively.

The fatty acids increase in solubility as the series is ascended, up to a certain point, when the solubility apparently reaches a maximum, and then slowly decreases. Formic acid is only slightly soluble, and it is hoped that this acid may be separated from the other lower fatty acids by shaking out an aqueous solution with the various solvents. The solubility of the higher acids is also being studied.

As with trichlorethylene, the dicarboxylic and hydroxy acids are insoluble. The sparing solubility of cinnamic acid is interesting, and should prove useful for separating this acid from other aromatic acids. Phthalic, salicylic, and mandelic acids are soluble in the hot solvent, but crystallise out on cooling. The esters are all soluble, and, as would be expected, the carbohydrates are all insoluble. The solubility of the glucosides evidently depends on their constitution.

Iodoform, when warmed with either tetra- or penta-chlorethane or tetrachlorethylene, is decomposed, liberating iodine, which goes into solution. In dichlorethylene and trichlorethylene it dissolves without decomposition.

Acetamide appeared to be volatile in the vapours of tetrachlorethylene. Other amides are being studied.

The solubility of caffeine in dichlorethylene suggests a method for separating it from other purine derivatives.

The amino-acids are being studied more fully. Practically all bases are soluble, but their salts are insoluble.

The solubility of the osazones and hydrozones is being studied further.

The solubility of the gums and resins is only roughly indicated. The matter is being investigated quantitatively.

The solubility of the alkaloids is being specially studied at length, as useful separations are anticipated.

The solubility of forty-five dyes has been tried. The following tables indicate what sort of separations might be expected. Much work has yet to be done, and it is anticipated that the tables will be modified somewhat later.

TABLE FOR THE EXTRACTION OF THE DYES.

Extract with C_2Cl_4.

Soluble [A.].
Nigrosin (Spirit sol.).
Victoria Blue (B).
Leaf Green.
Ethyl Green.
Rhodamine (B extra).
Sudan III.

Insoluble.

Extract with C_2HCl_5.

Soluble [B.].
Victoria Blue (BS).
Aniline Blue (II B).
Spirit Green (II).
Methyl Violet (B).
Methyl Violet (B extra).

Insoluble.

Extract with C_2HCl_3.

Soluble [C.].
Aniline Blue (BB).
Chrysoidine.
Malachite Green.
Diamond Green (G).
Diamond Green (B).
Rosaniline.
Metanil Yellow (extra).
Auramine (OO).
Eucrysine (RR).

Insoluble.

Extract with $C_2H_2Cl_4$.

Soluble [D.].
Japan Black.
Jet Black (BT).
Patent Blue (A).
Neptune Blue.
Rubin (N).
Saffranine Scarlet (B).
Saffranine Scarlet (G).
Phenolphthaleïn.

Insoluble.

Extract with $C_2H_2Cl_2$

Soluble [E.].
Nigrosin (G).
Methylene Blue.
Vesuvine (OOOL).
Spirit Scarlet (G).
Indulin Scarlet.
Bismarck Brown.

Insoluble [F.].
Nigrosin (Water sol.).
Methyl Blue.
Soluble Blue (I N).
Nicholson's Blue.
Vesuvine (BL).
Eosin (Yellowish).
Eosin (Bluish).
Cotton Scarlet.
Indigo Carmine.
Methyl Orange.
Fluorescein.

[A].

Extract with C_2HCl_5.

Soluble.	*Insoluble.*
Leaf Green.	Nigrosin (Sp. sol.).
Rhodamine (B extra).	Victoria Blue (B).
Sudan III.	Ethyl Green.

Extract with $C_2H_2Cl_4$.

Soluble.	*Insoluble.*
Nigrosin (Spirit sol.).	Ethyl Green.
Victoria Blue (B).	

[B].

Extract with C_2HCl_3.

Soluble.	*Insoluble.*
Victoria Blue (BS).	Aniline Blue (II B).
Spirit Green (II).	
Methyl Violet (B).	
Methyl Violet (B extra).	

[C].

Extract with $C_2H_2Cl_4$.

Soluble.	*Insoluble.*
Aniline Blue (BB).	Chrysoidine.
Malachite Green.	Rosaniline.
Diamond Green (G).	Metanil Yellow (extra).
Diamond Green (B).	
Auramine (OO).	
Eucrysine (RR).	

[D].

Extract with $C_2H_2Cl_2$.

Soluble.	*Insoluble.*
Japan Black.	Saffranine Scarlet (G).
Jet Black (BT).	
Patent Blue (A).	
Neptune Blue.	
Rubin (N).	
Saffranine Scarlet (B).	
Phenolphthaleïn.	

For the compounds already tried the following rules for solubility hold good: (1) If a compound contains only one hydroxyl group, it is generally soluble. (2) If, however, it contains two or more hydroxyl groups, or one hydroxyl and one carboxyl, or two or more carboxyl groups, it is more or less insoluble. (3) Ortho compounds are generally more soluble than the meta or para compounds. (4) The solubility of a compound generally decreases as the chlorine contents of the solvent increase. (5) An accumulation of nitro groups tends to decrease solubility.

USES IN ANALYSIS.

Fat Estimations.—In the author's paper on trichlorethylene (*loc. cit.*) it was proposed to use trichlorethylene for fat estimations in various substances, and it was shown that results comparable with those given by ether could be obtained if the substance was first dried and certain precautions were observed. Since then R. Neumann (*Chem. Zeit.*, 1911, **35**, 1025-1026) has proposed to extract with trichlorethylene by shaking in the cold, filtering, and evaporating down an aliquot part of the filtrate and weighing. G. Grimme (*Chem. Rev. Fett- u. Harz-Ind.*, 1912, 191-195) has compared the estimations of fat made by cold extraction with various chlorhydrocarbons with those obtained with ether. He gives the following average results:

Ether.	$CHCl_3$.	CCl_4.	$C_2H_2Cl_2$.	C_2HCl_3.	C_2Cl_4.	$C_2H_2Cl_4$.	C_2HCl_5.
7·45	8·58	7·43	8·13	7·46	7·79	7·71	9·62

Only in the case of carbon tetrachloride and trichlorethylene did the fat agree in amount, colour, etc., with that extracted with ether. He describes a modification of Neumann's method. D. P. van Lennep and J. D. Ruys (*Chem. Weekblad.*, 1912, **9**, 654-657) record results of a number of extractions of milk fat with trichlorethylene, and recommend it on account of its non-inflammability. R. Neumann (*Landw. Versuchs-stat*, 1913, **80**, 701-736) considers that the cold extraction with trichlorethylene has advantages over ether, inasmuch as the substance need not be previously dried. This is directly opposed to what the author found (*loc. cit.*); and after careful study of the matter he still considers it preferable to dry before extraction, as traces of water in the trichlorethylene cause it to decompose more readily in the light, forming hydrochloric acid, which acts on the fat. This, of course, would not be so necessary for a short extraction in the cold.

Experiments were made to construct an apparatus for use with trichlorethylene, making the use of cork or ground-glass joints unnecessary. Mercury is attacked, so mercury seals were out of the question. Finally, it was decided to modify the apparatus proposed by C. Beadle and H. P. Stevens (ANALYST, 1913, **38**, 143-144), so that the flask might be directly weighed. The details of this apparatus will be described in a future communication.

The apparatus has been found to be very efficient for various extractions at the boiling-point of the liquids. There is absolutely no loss with dichlorethylene, which has the lowest boiling-point (55° C.), while trichlorethylene is condensed almost entirely upon the base of the condenser. Experiments with this apparatus for the determination of fat are still in hand, but it is anticipated that an apparatus in which the thimble is kept quite cold will be necessary. The good results obtained by the author previously are ascribed to the fact that a very large extractor with ground-glass joints was used, and the liquid in the Soxhlet kept comparatively cold.

Indiarubber Analysis.—The apparatus is being used for extracting rubber with the solvents. Conclusive figures are not yet available, but it is indicated that for a sample of vulcanised rubber the dichlorethylene extract is about equal to the acetone extract *plus* alcoholic extract. The chlorethanes have been used to extract the rubber from soft varieties in order to determine protein matter, etc.

The Separation of the Higher Saturated from the Unsaturated Acids.—The solubility of the lead salts of palmitic, stearic, and oleic acids have been determined qualitatively, and, as would be expected, lead oleate was extremely soluble; while in the cold, lead palmitate and lead stearate were relatively insoluble.

Lead palmitate and stearate were, however, soluble when hot, and crystallised out almost completely on cooling. Lead stearate was much more soluble than lead palmitate when hot. In view of this, other salts are being investigated, and the solubilities determined quantitatively.

Use in Determination of Iodine Value of Oils.—As bromine is rapidly absorbed by the chlorethylenes in the light, it was expected that iodine would also be absorbed in a similar manner. To test this point, a few c.c. of each solvent were placed in test-tubes, and about double the amount of Wijs' solution was added. The test-tubes were placed on a window-sill facing due south, in full sunlight. A very slow absorption took place after four days; the liquids began to turn pale on the top with the exception of the dichlorethylene, which did not appear to alter. As this seemed fairly hopeful, the following quantitative experiments were made: Ten c.c. of each solvent were placed in bottles as usually used for iodine absorptions; 25 c.c. of Wijs' solution were added, and they were then put into the cupboard for seventeen hours. At the end of this time 10 c.c. of potassium iodide solution were added, and the iodine titrated with $\frac{N}{10}$ sodium thiosulphate, using starch as indicator, in a place out of the direct rays of the sun. The results are given below:

Carbon tetrachloride	43·85 c.c. (control)
Dichlorethylene	42·00 41·90
Trichlorethylene	43·90
Tetrachlorethylene	43·90
Tetrachlorethane	43·90
Pentachlorethane	43·80

It will be noticed that the agreement with carbon tetrachloride is within experimental error, with the exception of dichlorethylene, which comes very low. The experiment was repeated in this case, and confirmed the previous figures. It is remarkable that in the qualitative experiments the dichlorethylene behaved differently to the others. This might be due to the fact that it is a mixture of two isomers.

As these chlorine derivatives are now used extensively for various purposes, it is not uncommon to have to examine substances containing them. These substances can be divided into two classes. Firstly, where they are added as adulterants; and secondly, where they form one of the chief ingredients in such things as rubber solutions, paints, varnishes, etc.

They are fairly easily recognised by their odour, and, when isolated in the pure state, the physical constants given will determine which member is present, or will indicate a mixture. If a mixture is being dealt with, the addition of bromine will indicate chlorethylenes.

The action of aluminium trichloride is useful, as it apparently only reacts with the chlorethanes and tetrachlorethylene.

The chlorethylenes form salt-like compounds with alkaline mercuric cyanide, the ethylene linking remaining intact (Hoffmann and Kirmeuther and A. Thal, *J. Chem. Soc.*, 1910, A I., 168). The chlor compounds are simply shaken with the alkaline mercuric cyanide solution. The trichlorethylene compound melts at 83° C.

An interesting reaction apparently takes place between pentachlorethane and phenylhydrazine at ordinary temperatures, when simply mixed and allowed to stand. A copious evolution of gas takes place, and a solid compound is formed which, when washed with petroleum ether (in which it is insoluble) to free it from excess phenylhydrazine or pentachlorethane, is of a dark golden colour. This compound and the action of phenylhydrazine on the chlorethylenes is being more fully investigated.

In conclusion I have to thank Mr. McLachlan, who has given me valuable assistance; also Mr. McKenzie and Mr. H. S. Constantine, who have given useful help in connection with certain parts of the work.

The Weston Chemical Co. kindly supplied the solvents for the experiments.

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THE EXAMINATION OF COMMERCIAL GELATINS WITH REGARD TO THEIR SUITABILITY FOR PAPER-MAKING.

By R. W. SINDALL and W. BACON, B.Sc., F.I.C.

(*Read at the Meeting, November* 5, 1913.)

The examination of gelatin presents many difficulties owing to the complex nature of its constitution. The ease with which the true gelatin hydrolyses to albumoses and peptones, not only under chemical and bacterial action, but even under the influence of heat and moisture alone, renders the task somewhat difficult in differentiating the class of stock from which such gelatins have been prepared. It is true that gelatin when kept dry is moderately stable even after a considerable period, and permits the determination of certain physical properties, which, if properly carried out, gives a sufficiently true indication of the extent of hydrolysis. It must be borne in mind that owing to the soluble nitrogenous constituents of gelatin, the latter is rendered extremely liable to putrefactive and hydrolytic changes under the influence of liquefying bacteria, especially in warm weather.

For paper-making purposes we carry out the analysis of a sample of gelatin as follows:

Ash.—Ten grms. of the gelatin are carefully charred, extracted with boiling distilled water, and filtered. After repeating this two or three times in order to remove the soluble salts, which consist for the most part of chlorides, the carbonaceous mass is burnt, the filtrate is added and evaporated to dryness, and, after heating gently, it is weighed. If the percentage of ash is below 2 per cent., no further examination of the ash is made. When above this limit, the ash is fused with fusion mixture and a little nitre, if the presence of chromium be suspected; the silica is separated in the usual manner, and the filtrate treated with ammonia. If any precipitate be obtained, this will probably consist of alumina and iron, both free and combined with phosphoric acid, together with a trace of lime. The alumina precipitate is thoroughly washed, ignited, and weighed. The filtrate is made up to a known bulk, and aliquot portions taken for the determination of lime, magnesia, and sulphates. Further examination of the alumina precipitate is carried out upon another 10 grms. of the gelatin, the method followed up to the precipitation of the alumina being exactly similar. After thorough washing, the precipitate is placed in a beaker, dissolved in pure nitric acid, and made up to 100 c.c.; 25 c.c. are precipitated with ammonium molybdate reagent, and the solution kept at a temperature of about 60° C. for six hours. The precipitate is filtered and washed with ammonium nitrate, and finally with 1 per cent. nitric acid.

The lime is determined as oxalate after adding citric acid, ammonia, and finally boiling with acetic acid. It is very rare to find any lime in the ammoniacal precipitate. The iron is determined colorimetrically against a standard solution of iron, and the alumina is thus found by difference.

The question may be raised as to the importance of determining so carefully the amount of alumina. Since alum or sulphate of alumina precipitates chondrinogen and mucin, it is quite conceivable that such a salt would be used in aiding the clarification of the gelatin liquors. More important still to the paper-maker, however, is the fact that many gelatins are bought on a standard in which the gelatin should have a specified jelly strength, or "tenacity." Now, sulphate of alumina increases this physical factor, so that by the addition of this salt a totally fictitious result is obtained, as is shown by the experimental figures obtained with two samples of gelatin given in Tables I. and II.

It is important, therefore, in judging the value of a paper-maker's gelatin, to determine whether the tenacity is dependent solely upon the hydrolysed fraction of the collagen—namely, gelatin proper—or whether such an apparent strength is brought about by the use of alum.

Chlorine and Chlorides.—The examination of gelatin for chlorine, or, more properly, chloroamines, and in a lesser degree chlorides, is most important. The presence of chloroamines in gelatin affect the physical properties, especially on storage, by reducing the strength and toughness of the film or cake. From a bacteriological point of view, owing to the inhibiting action of the chloroamines upon bacteria, they might be regarded as desirable, but any advantages in this direction are more than counterbalanced by their liability to lower the strength of the gelatin. The test can be made with iodide of potassium and starch, slightly acidifying with acetic acid. A blank test should be carried out on the same chemicals.

TABLE I.

RELATION OF STRENGTH OF GELATIN TO PERCENTAGE OF ALUM.

Strength of Gelatin 5 per Cent. in all Cases.

SAMPLE A.

No.	Alum per Cent. of Gelatin.	Sinking Weight. Temperature 15° C.	Sinking Weight. Temperature 22° C.
		Grms.	Grms.
Blank.	0	29·0	Nearly fluid.
1	1	30·0	18·2
2	2	40·5	20·8
3	3	69·0	33·1
4	4	72·5	34·0
5	5	93·5	34·7
6	6	99·0	42·3
7	7	96·2	41·6
8	8	96·7	36·5
9	9	93·0	25·6
10	10	85·0	26·3

TABLE II.

Strength of Gelatin 5 per Cent. in all Cases.

SAMPLE B.

No.	Alum per Cent. of Gelatin.	Sinking Weight. Temperature 15° to 16° C.	Sinking Weight. Temperature 21° to 22° C.
		Grms.	Grms.
Blank.	0	4·50	Liquid.
1	1	6·30	3·67
2	2	8·50	5·80
3	3	11·20	8·20
4	4	15·55	10·00
5	5	17·03	9·90
6	6	17·70	9·90
7	7	22·00	13·50
8	8	19·80	6·40
9	9	19·60	4·50
10	10	18·80	7·80

Chlorides act similarly, but these possess the additional disadvantage of being more or less hygroscopic, and so tend to render the paper sized with a gelatin containing a high proportion of these salts more liable to putrefactive changes.

Ten grms. of gelatin are heated on a bath with distilled water acidified with nitric acid, and filtered if necessary from any traces of connective or epidermal tissue. Silver nitrate is added, the solution brought to the boil, and allowed to stand all night. If care be taken, the clear liquid can be withdrawn almost completely by means of a siphon. Distilled water and a few drops of ammonia are added to redissolve the precipitate; it is then boiled, and the silver chloride reprecipitated by means of nitric acid. Treated in this manner, the precipitate is obtained free from empyreumatic substances which are occluded in the first precipitation.

Moisture.—An average sample of the gelatin is filed down until 5 grms. of the dust pass through a sieve having 70 meshes per linear inch. The air-dried gelatin is placed in a Schleicher and Schull thimble, previously dried and weighed, and allowed to stand for from eighteen to twenty-four hours over sulphuric acid. When treated in this way, the gelatin loses about 6 per cent. of moisture. Owing to this preliminary drying, when the powdered gelatin is put into the oven for complete drying no adhesion of the particles takes place. This perhaps is not so important for the actual determination of moisture; but as the gelatin is subsequently used for the determination of fat, the advantage of such a system of drying is obvious. The amount of moisture varies, and usually lies between 11 and 14 per cent.

Fat.—After drying, the gelatin is extracted with dry ether in a Soxhlet for eight hours after allowing to soak all night in the solvent at the room temperature. It is as well in determinations of fat, owing to the very low percentages of this constituent (sometimes below 0·05 per cent.), to weigh the flask against another tared flask, and to re-extract the ethereal extract with two or three successive small quantities of ether; after drying the flask, the difference will be the amount of fat. The reason for this is that after hot extraction of the gelatin for eight hours, some of the glue substance other than fat is dissolved or mechanically carried over. By such a method as described above, this error is avoided.

Tenacity or Strength of Jelly.—Twelve and a half grms. of gelatin are soaked in 237·5 grms. of water over night, and in the morning warmed just sufficiently to bring the whole into solution. It is then cooled down until it will just flow, and poured into a large beaker. In this way the skin which is always formed by evaporation of the solution is practically avoided. A definite temperature is fixed upon—namely, 15° C.—and any departure from this either above or below makes, relatively, a considerable difference in the jelly strength. After about six hours' setting the surface is abraded, and a tube having a bulb of $\frac{1}{2}$-inch diameter blown at the lower end is placed with the bulb upon the surface, mercury being poured in from a burette, lifting up the tube while each addition of mercury is made. The addition of mercury is made at such a rate that the operation of forcing down the bulb to $\frac{1}{2}$-inch from the bottom of the beaker or tumbler takes five minutes.

It is obvious that in carrying out this test, although dependent upon the same physical property—namely, surface tension—different factors come into consideration—for example, size and form of bulb, and material of which the bulb is made. It is, therefore, necessary that each operator should standardise the instrument he uses on standard gelatins.

Viscosity Test.—This test is one of the most important from a paper-making point of view, for although strength of jelly is correlated with high viscosity, a point is reached when high jelly strength must be subordinated to penetration. Owing to the osmotic character inherent to fibres—*i.e.*, the tendency to separate out the colloidal substance from the crystalloids—it is obvious that solutions possessing high viscosity must be slower in penetration, and so allow the walls of the fibre to exert osmosis. The net result is—and this is borne out in practice—that papers sized with such a solution of gelatin would hold too great a proportion of the gelatin on the surface.

For this test we use the type of viscometer suggested by Dr. Fernbach in his book on "Glues and Gelatins," with a slight modification as to the orifice, which is so drawn off and standardised that 50 c.c. of water at 60° C. passes through in exactly fifteen seconds.

A 25 per cent. solution of gelatin is heated in a flask to 60° C. The solution is then sucked up in the pipette, which, being fitted into an upright condenser tube, can easily be maintained at the required temperature by means of any convenient water heater. From the lower end the rod which closes up the orifice of the upright condenser is removed and the pipette pushed down to a point just below the cork. The tap fitted to the upper part of the pipette is opened, and the solution allowed to drip slowly until it reaches the 50 c.c. mark. It is then allowed to fall freely, a stop-watch started at the same instant, and the time recorded.

We would point out here that where many determinations are made every day, there is a gradual enlargement of the orifice, owing to the friction of the colloidal solution. A standard instrument should be kept, and the viscometer checked against the standard instrument at least once a month.

Free and Combined Sulphurous Acid.—Ten grms. of the gelatin are soaked for a short time in the cold in an ordinary distillation flask, 10 grms. of phosphoric acid (sp. gr. 1·7) are then added, the whole heated in a water-bath under a condenser until the gelatin is completely hydrolysed. The bath may then be removed and the heating continued over a gauze without fear of breaking the flask. We have found in practice that when 250 c.c. have distilled over, all the sulphurous acid is in the distillate. A drop of $\frac{N}{10}$ iodine solution is placed in the trap to insure the retention of the whole of the SO_2.

The distillate is divided, one-half titrated with $\frac{N}{10}$ iodine solution, and the other half, using phenolphthaleïn as indicator, with $\frac{N}{10}$ caustic soda. The difference between the two numbers will be a measure of any volatile acid, such as acetic acid, that may be present.

Chondrinogen Test.—We have termed this the "chondrinogen test" with a certain amount of reservation. When a solution of gelatin, apparently quite clear or opalescent after filtration from any actual sedimentary tissue, is heated for some time with 5 per cent. nitric acid, the somewhat colloidal character of the solution lessens, and there is a gradual coagulation of matter. After filtering off and washing, the precipitate is dissolved in $\frac{N}{10}$ caustic soda, the solution becoming much yellower in colour. The solution thus obtained is treated with acetic acid in slight excess, when complete precipitation occurs. In large excess, however, re-solution takes place. Tannic acid and alcohol also cause precipitation. It has not the slimy character of mucin, though, like this substance, it reacts with sulphate of alumina.

We are continuing our investigations on this body to see if the presence of such a compound bears some direct relation to the use of low-grade stock in the manufacture, such as would be evidenced in the viscosity and jelly strength, or to an imperfect method of manufacture. It is quite obvious that since the paper-maker uses alum with his gelatin, the production of such a compound as shown is strongly to be deprecated, and we have, working on these lines, made a quantitative test of this reaction. Ten grms. of the gelatin are heated with 200 c.c. of 5 per cent. pure nitric acid at 60° C. until the opalescent or cloudy solution becomes perfectly bright. It is then filtered off on to a weighed filter-paper, washed, dried, and weighed.

On ordinary gelatins as used by the paper-maker, results have been obtained as under:

0·54 per cent.
0·45 ,,
0·28 ,,
0·24 ,,
0·20 per cent.
0·16 ,,
0·12 ,,
0·09 ,,

Precipitates with alum were obtained in the first two samples.

DISCUSSION.

Dr. R. L. FERNBACH said that ultimately there must be some method developed for determining whether in a given glue or gelatin there was present any stock, or any material representing stock, which had failed to respond to the natural hydrolysing processes of manufacture, and, furthermore, that systematic research would in the long run establish some relationship between the presence of such unhydrolysed materials and the actual strength of the glue or gelatin from a commercial standpoint. It seemed to him that the magma which the authors found to be formed on treatment of the gelatin solution with nitric acid was the result of imperfectly hydrolysed stock. This matter, which was of considerable interest, had been studied by him in connection with some analyses which he had made on behalf of the Department of Agriculture at Washington. In the United States gelatin was debarred by law from use in food if it could be shown that it was undergoing putrefaction, or—which was more difficult—if it could be shown to have been made from stock which had undergone putrefaction. To say that there were any well-defined analytical methods which would enable one to certify that gelatin or glue had been made from stock undergoing putrefaction would be to make too sweeping a statement; but if analytical results were combined and studied in conjunction with practical works' experience, some very interesting inferences might be drawn. In the cases to which he was referring, the product was made from South American stock, which reached the manufacturer in an advanced state of putrefaction. It required most careful treatment, and it was hardly necessary to mention that, owing to the putrefaction, much of what would have been valuable glue-making material was wasted. Of course, with the aid of works' experience it was possible to determine from what stock a glue had been made; but he preferred to digest the glue with sulphate of alumina instead of with normal alum, and he found that after a short period of digestion a precipitate was obtained similar to that referred to by the authors. The method which they described appeared, however, to afford a shorter and more accurate means of determining the presence of unhydrolysed stock; but, as they had mentioned, it still remained to be shown whether the proportion of this had any relation to the strength of the product. Even, however, if there were no such relationship, it would seem, from the point of view of the paper manufacturer who demanded a product of a definite degree of purity, that the test when fully developed would prove valuable.

Mr. BEADLE said that one of the most practical methods used by the paper-maker for testing gelatin was to make up, say, a gallon of jelly from each sample, the proportions being adjusted, on the basis of the respective prices, so that the cost of each gallon was the same. As a rule it was found that the sample yielding the stiffest jelly was the best for the purpose. Often its price was higher than that of others, the quantity used being therefore less; but it was more a question of the stiffness of the jelly than of the proportions of the ingredients. It was very important that the gelatin should be in the form of jelly in the paper itself, prior to drying—that is, immediately after it had passed through the sizing trough and the squeezing press. The reason of this was that if the paper was dried at a temperature at which the gelatin remained liquid there was a tendency merely to coat the fibres; but if the conditions of drying were such that the gelatin first of all set into a jelly, the minute spaces between the fibres became, as it were, bridged over and filled up. Since some heat was needed to give the air sufficient drying power, the lower the temperature at which a 5 per cent. solution set to a jelly, the more valuable the gelatin would be from a paper-maker's point of view.

Dr. RIDEAL remarked that in the case of the viscometer with which he had worked (which was a modification of the Slotte instrument), the results could be calculated into absolute units, and this seemed to him to be an advantage over the pipette form. The only difficulty lay in the orifice, or rather the tube at the end, which was of a definite length, namely, 10 c.m., and of a fixed known diameter, while the volume of the bulb was 150 c.c. The guard bulb at the top was very useful, making it quite easy to note the exact time at which the reading was begun, this being somewhat difficult with the pipette closed by the finger. With a liquid of high viscosity, the time taken for a test could be reduced by working under diminished pressure.

Dr. FERNBACH said that he had used the Rideal-Slotte viscometer with excellent results. In glue testing, any form of viscometer could be used, but the objection to most of them was that the time required for the solution to run through was too long—an important consideration when the daily number of samples was large. There was, of course, no great scientific novelty about the pipette form of instrument, but he did not think that the objection mentioned by Dr. Rideal held good, for he had never found any difficulty in starting the stop-watch at the exact moment at which the pipette was opened.

Mr. J. H. B. Jenkins asked whether there was any fairly uniform relationship between the viscosity and the jelly strength. In other words, was it really necessary to determine both these factors, or could the one be roughly deduced from the other?

Mr. T. Macara asked whether the authors had observed any relationship between the strength of the jelly and the amount of water absorbed by the gelatin in the cold, which was considerable. Personally he had never been able to establish any such relationship.

Mr. H. F. Knight asked if the authors would explain exactly what was meant by "standard gelatin."

Mr. Bacon, in reply, said that there seemed no doubt that the jelly strength and the viscosity were co-ordinated, a high jelly strength being always accompanied by a high viscosity. He had not, however, up to the present, been able to determine exactly what the ratio between them was: it never seemed to be quite the same. The paper-maker, of course, desired a maximum of strength with the minimum of viscosity. In the case of food gelatin they had met with samples which absorbed as much as sixteen times their weight of water, but with gelatin for paper-making the absorption after forty-eight hours was usually about six times the weight of the gelatin. The quantity of water absorbed, however, was of less importance than the condition of the gelatin after treatment. A satisfactory sample, after forty-eight hours' soaking, would still retain its properties of toughness and absence of tendency to break; whereas a sample of bone glue, on similar treatment, would show a slimy surface, owing probably to the high proportion of peptones and albumoses, some of which passed into solution. The exhibition of a slimy surface after soaking was in itself a sufficient ground for rejection. Their use of the term "standard gelatin" was perhaps somewhat loose. Dr. Fernbach used what was called the "Cooper Grade" standard, but in this country one had to make one's own standard, which, however, was easily done by selecting samples showing a high strength together with a suitable viscosity.

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NOTES ON THE INSOLUBLE BROMIDE TEST FOR DRYING OILS.

By JOHN A. L. SUTCLIFFE, A.I.C.

(*Read at the Meeting, December* 3, 1913.)

The great variation in the values obtained by different workers for the amount of insoluble brominated glycerides resulting from the bromination of linseed oil has tended to deprecate what is really a most valuable test for drying oils. In the case of linseed oil, values have been published varying from 23 to 50 per cent., and it has been suggested that the chief value of the test is to enable a chemist to judge, from his own previous experience of the test, as to the interpretation of the results he himself obtains for any particular oil. It has also been suggested that the difficulty is best got over by the bromination of the fatty acids obtained from the oil. The investigation of which the following is a description, was undertaken with the object of finding the reason for these variations, and of arriving at a definite method of performing the test.

To determine if the purity of the ether used in the test was a matter of importance, some ether was prepared by washing methylated ether (sp. gr. 0·720) three times with water, drying over calcium chloride, and distilling. The distillate was dried over metallic sodium and again distilled. For the series of comparative tests, a linseed oil of iodine value 185 was used.

One grm. of the oil was weighed out into a small tared Soxhlet flask and dissolved in 40 c.c. of ether, 5 c.c. of glacial acetic acid were added, and the flask and contents cooled in running water to about 11° C. Bromine was added, drop by drop, with constant shaking until excess was denoted by the *red* colour of the solution. The flask was corked and allowed to stand in running water all night. The contents were filtered through two tared, superimposed filters, the precipitate being washed with four successive portions of 10 c.c. of ether. The filters and flask were dried in the water-oven for two hours and then weighed. Percentage of bromide = 38·7; 38·9. On repeating the experiment, using ordinary redistilled ether, the values obtained were 38·8, 38·4, 38·9, 38·8 per cent.

As the ether would therefore seem to have no effect, the rest of the experiments were carried out with redistilled ether.

The experiment was repeated, but in this case the flasks were only allowed to stand for three hours. The results obtained were—37·8, 38·07, 38·3, per cent. Although the difference in this case was small, it was found on examining other oils that in many cases a considerable amount of precipitate separated after three hours and up to twenty-four hours. The rest of the determinations were made by allowing to stand overnight, except in the cases instanced below.

The instructions to wash with three 10 c.c. portions of ether were not adhered to, as it was found that in most cases the washing was insufficient. It was also found that if the ether used for washing were not cooled, the results obtained were always low, and that it was possible to recover insoluble bromides from the washings by concentrating and cooling. As the original instructions require the precipitation to take place at a temperature approaching 0° C., and also require the bromine to be added till the solution is brown and not red, this method was tried with the following results :

	Three Hours on Ice.	Twelve Hours on Ice.
Bromine added till red	36·9, 37·4	37·4, 37·8
Bromine added till brown ...	23·2, 25·1, 27·5	24·7, 28·2

It will be seen that better agreement is obtained by the former method of allowing to stand in running water.

The effect of the amount of acetic acid was tried. Using 10 c.c. of acid in place of 5 c.c., the above oil gave 29·4, 29·9, instead of 38·8 per cent. The method of filtering by suction through chamois leather, recommended by Hehner and Mitchell (ANALYST, 1898, **23**, 310), gave results which agreed badly, and it was abandoned.

The method recommended in view of the foregoing considerations was the following: One grm. of oil is taken in a tared flask and dissolved in 40 c.c. of redistilled ether, 5 c.c. of glacial acetic acid are added, and the flask and contents cooled to about 11° C. in water. Bromine is added drop by drop with constant shaking till the excess is denoted by the red colour of the solution, and the flask is corked and allowed to stand overnight in water. The contents of the flask are filtered through a Gooch crucible prepared in the ordinary way with a small circle of filter-paper fitted over the asbestos. This enables the bromides to be separated for the melting-point determination. The precipitate is kept as much as possible in the flask during the first three washings with 10 c.c. quantities of cooled ether, and is then transferred to the Gooch crucible for the two final washings. The Gooch crucible and flask are dried for three hours in the water oven, and are weighed. After weighing, the melting-point of the bromides is determined. In the case of linseed oil, this should be from 141° to 144° C.

The values for several oils were determined by the above method, with results as under :

	Iodine Value.	Percentage Bromide.
Baltic oil	204	49·3
,, ...	199	47·0
Oil of unknown origin	197	46·8
,, ,, ,, ...	194	44·0
,, ,, ,, ...	192	42·6
Bombay oil	190	41·6
Oil of unknown origin	188	40·6
,, ,, ,, ...	187	39·7
,, ,, ,, ...	185	38·8
,, ,, ,, ...	184	37·9
,, ,, ,, ...	180	35·2
Plate oil (cold pressed in laboratory) ...	179	34·6

If a curve is plotted of these values against the iodine value, a straight line results, which may be expressed by the formula : Per cent. of bromide = (0·63 iodine value) − 78·0.

If the bromine values are calculated from the iodine values and the curve for the bromides plotted against them, it will be found that the formula for this curve is—Per cent. of bromides = bromine value − 78·0.

This would seem to suggest that the gradient of the curve is correct, and that variations in the conditions of the experiment would affect the constant 78 and not the factor 0·63. The above method seems to give results sufficiently capable of interpretation to justify the belief that the determination of the values on the separated fatty acids is in most cases unnecessary. More work is being done on the chemical problems suggested by, and arising from, the above experiments, and on the application of the process to blown and oxidised oils of various types. The result of these investigations will form the subject of a later communication. I have to thank Dr. Harry Ingle of Leeds for valuable criticism and advice, and for several specimens of oils.

City of Birmingham,
Analytical Department.

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THE USE OF THE EXPRESSIONS "COCOA BUTTER" AND "COCOANUT OIL."

By Professor C. A. NEUFELD, Ph.D.

The writer desires to draw the attention of English chemists to the similarity in the way of writing cocoa butter "the fat of the seeds of the cocoa-tree" (*Theobroma cacao*), and of cocoanut oil "the fat of the cocoanuts" (*Cocos minifera var. butyracea*). As the butter fat is termed "kokosfett" or "kokosbutter" in the German language, confusion frequently arises when English chemical journals are abstracted by German writers.

J. Lewkowitsch, in the German edition of his well-known book "Oils, Fats, and Waxes," wrote cacao butter instead of cocoa butter, and he pointed out that he did so to avoid confusion. H. W. Wiley in "Foods and their Adulteration" uses the terms "cacao butter" and "cocoanut oil or butter."

It would be desirable, and also much appreciated by their international colleagues, if English chemists would adopt the expressions "cacao butter" and "coconut oil or butter" as applied to the products of *Theobroma* and *Cocos* respectively.

Kgl. Untersuchungs-Anstalt
für Nahrungs- und Genussmittel
zu Würzburg.

[Professor Neufeld has undoubted grounds for bringing this matter to the notice of English chemists. The Publication Committee of the Society has decided to adopt his suggestion.—Editor.]

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

Solvents for Alkaloids and Alkaloid Salts. G. L. Schaefer. (*Amer. J. Pharm.*, 1913, **85**, 439-442; through *Chem. Zentralbl.*, 1913, II., 1828-1829.)—In investigating the solubilities of the principal therapeutic alkaloids and their salts, the author found that the addition of methyl alcohol considerably increases the solvent action of other organic media. For the estimation of morphine in its preparations, a quantity of the material equivalent to about 0·3 grm. of morphine is rubbed to a fine paste with washed sand, excess of sodium bicarbonate, and a little water; this is dried at 30° to 40° C., and digested for an hour with 30 to 40 c.c. of a freshly prepared mixture of 1 part of methyl alcohol with 4 of chloroform or benzene. After filtering, the residue is washed three or four times with 10 c.c. of the solvent; the extract is evaporated, and the residue treated with excess of $\frac{N}{20}$ sulphuric acid, the acid then being titrated back. This method is not suitable for the extraction of morphine from liquids, for which purpose a mixture of equal parts of chloroform and the fraction of fusel oil distilling between 90° and 110° C. is recommended. This solvent does not emulsify; 1 part of morphine is soluble in 60 parts of the warm solvent, and in 140 parts of the cold. A table of solubilities of several alkaloids and salts in various solvents and mixtures is given. J. F. B.

Estimation of Caffeine in Kola Preparations. M. François. (*J. Pharm. Chim.*, 1913, **8**, 411-422; 451-458.)—An investigation of the estimation of caffeine in kola-nut preparations (extracts) indicated that the most trustworthy results may be obtained by simply treating the material with water and magnesia, drying the mixture, grinding it with sand, and extracting the mixture with chloroform. The caffeine is extracted completely, is free from ash, and does not contain more than 3 per cent. of its weight of substances which are·insoluble in hydrochloric acid. This method of estimation may be applied to preparations containing sugar. In the case of ground kola-nuts, however, the process yields low results owing to the fact that the magnesia does not penetrate the tissue sufficiently to liberate the whole of the caffeine. Further investigation in this direction is required. W. P. S.

Estimation of Morphine in Acid Opium Solutions. Debourdeaux. (*J. Pharm. Chim.*, 1913, **8**, 424-429.)—The following method may be used for the estimation of morphine in such preparations as Sydenham's laudanum (a tincture of opium flavoured with saffron) and of "black drops" (*Acetum opii crocatum*), and is particularly useful in the case of preparations containing free acetic acid. One hundred and fifty grms. of the solution under examination are treated with an excess of milk of lime, diluted to 300 c.c., and filtered. The insoluble portion is washed with 50 c.c. of water, then transferred to a beaker, diluted with 150 c.c. of water, filtered, and the insoluble portion again washed with 50 c.c. of water. The total filtrate is now saturated with carbon dioxide, evaporated to a small volume, the liquid and precipitate being then rinsed into a beaker and water added to bring the weight up to 100 grms. After the addition of 50 c.c. of ether and 10 c.c. of $\frac{N}{1}$ ammonia solution, the mixture is set aside for twenty-four hours, when the liquid is poured through a filter, and the crystals and filter are washed with five successive quantities of water saturated with ether and morphine. The filter and its contents are dried at 100° C., then placed in a beaker and mixed with milk of lime prepared by adding 5 grms. of slaked lime to 118 c.c. of water. When the morphine has dissolved, the mixture is filtered, and an aliquot portion of the filtrate is mixed with 10 per cent. of its volume of alcohol, its own volume of ether, and 2 per cent. of its weight of ammonium chloride. After standing twenty-four hours, the crystals which form are collected on a filter, washed with water saturated with ether and morphine, dried at 100° C., then washed with benzene, again dried, and weighed. W. P. S.

Characteristics of Stag's Fat. J. Klimont and E. Meisl. (*Monatsh.*, 1913, **34**, 1489-1492.)—A specimen of fat from the stag (*Cervus elaphus*) gave the following analytical values: Specific gravity at 50° C., 0·9066; melting-point (Pohl), 48° C.; solidification-point, 47·5° C.; acid value, 20·5; saponification value, 203·5; and iodine value, 19·3. When crystallised from hot acetone the fat yielded a mixed glyceride which melted at 62·5° to 63·5° C., and was identified as β-palmitodistearin. C. A. M.

Detection of Vegetable Fats in Animal Fats. M. Klostermann. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1913, **26**, 437.)—The method consists in the separation of cholesterol or phytosterol from the fats, and depends on the fact (*cf.* Windhaus, ANALYST, 1910, **35**, 256) that these two substances combine with digitonin to form a precipitate which is insoluble in alcohol and ether. In carrying out the test, 100 grms. of the fat under examination are saponified with 200 c.c. of 20 per cent. alcoholic potassium hydroxide solution; complete saponification is unnecessary, but the solution should remain clear when diluted with water. To the soap solution are now added 300 c.c. of water and 100 c.c. of 25 per cent. hydrochloric acid; the mixture is cooled and shaken with 250 c.c. of ether, this solvent being added before the fatty acids commence to solidify. The ethereal solution of the fatty acids is separated from the aqueous portion, washed three times with water, then diluted with 250 c.c. of petroleum spirit, and the mixture is well shaken after the addition of 25 grms. of sodium chloride. The solution is now filtered through cotton-wool, and to the warm filtrate is added 1 grm. of digitonin dissolved in 20 c.c. of alcohol. After fifteen minutes, the precipitate is collected on a filter, washed with ether until free from any trace of fat, then dried, removed from the filter, and acetylated by boiling it with 30 c.c. of acetic anhydride. The solution thus obtained is evaporated to dryness, the residue dissolved in 50 c.c. of alcohol, and water is added, drop by drop, to the solution until a precipitate begins to form; after a short time more water is added, so that the total volume introduced amounts to 25 c.c. The crystalline precipitate is collected, washed with 70 per cent. alcohol, and dissolved in ether. The ethereal solution is then evaporated, the residue is crystallised twice from alcohol, and the melting-point of the crystals is ascertained. Crystals obtained from various fats and oils and mixtures of the same had melting-points as follows: Lard, 110° C.; rape oil, 128° C.; lard + 2·5 per cent. of rape oil, 118° C.; lard + 10 per cent. of rape oil, 126° C. Poppy-seed oil, 128° C.; lard + 5 per cent. of poppy-seed oil, 121·5° C. Butter fat, 113° C.; cocoanut oil, 126° C.; butter fat + 2·5 per cent. of cocoanut oil, 115° C.

Since it is probable that cholesterol and phytosterol exist in fats partly in the form of their esters, and seeing that this combined portion of the two substances is not precipitated by digitonin, the author prefers to saponify the fat and to precipitate the cholesterol and phytosterol from the separated fatty acids instead of treating the fats directly with the reagent as proposed by Marcusson and Schilling (ANALYST, 1913, **38**, 458). W. P. S.

Detection of Gelatin in Sour Cream. A. Seidenberg. (*J. Soc. Chem. Ind.*, 1913, **5**, 927-928.)—Gelatin is usually detected in milk or cream by the precipitate it forms with picric acid after the removal of the proteins with acid mercuric nitrate. If, however, a milk or cream, showing no such precipitate when sweet, is allowed to go sour, and then tested, a perceptible turbidity is often noticeable. Since many samples are already sour when received for analysis, it is useful to be able to distinguish between the two precipitates (*U.S. Dept. Agri. Bull.*, **116**, 24).

The author finds that while both precipitates are soluble on heating in slightly acid solution, only the gelatin picrate is soluble in hot neutral water, and recommends that any precipitate formed with picric acid should be filtered off, washed with water containing 2 or 3 drops of ammonia per 100 c.c. until the washings are just alkaline to litmus. The washing is then continued with pure water, after which the precipitate is boiled and filtered while still hot. If gelatin picrate is present, it will pass through the filter, and may be again precipitated on the addition of an equal volume of picric acid. Sour cream containing as little as 0·5 per cent. of gelatin can be shown to contain this adulterant provided twenty or twenty-five c.c. of the sample are tested. H. F. E. H.

Quantitative Estimation of Gliadin in Flour and Gluten. G. A. Olson. (*J. Ind. and Eng. Chem.*, 1913, **5**, 917-922.)—It is usual to regard all the nitrogenous matter of flour which is soluble in dilute alcohol (50 to 70 per cent.) as gliadin. It is known, however, that if such a solution is evaporated and water is then added, a portion of the dissolved matter separates as a coagulum, and it is this portion alone which the author considers to be the true gliadin. There is a close agreement between the nitrogen in the alcohol-soluble coagulum and that obtained by adding together the nitrogen extracted by 1 per cent. salt solution and that dissolved by alcohol from the salt-extracted residue. The gliadin nitrogen obtained by this indirect method is much lower than that yielded by simple alcoholic extraction, but is in close agreement with the nitrogen found in the coagulum itself. Of the two methods for the estimation of gliadin, the coagulation method is the shorter and therefore more suitable to use than the indirect method. Alcohol of 50 per cent. by volume, as recommended by Hoagland (ANALYST, 1912, **37**, 19), has been adopted for the determination of gliadin nitrogen by the author, who finds that alcohols ranging between 50 and 65 per cent. by volume give similar results. Above 65 per cent. there is a rapid falling off in the nitrogen obtained as a coagulum. It was found that on the average about 68 per cent. of the total alcohol-soluble nitrogen is coagulable, or, in other words, is really gliadin; the other 32 per cent. is precipitated by phosphotungstic acid. From the same flours, as much nitrogen coagulum is obtainable from specially prepared dry gluten as from the flour itself. The easiest and shortest method for the estimation of the gliadin in flour is to deduct from the total nitrogen soluble in alcohol the nitrogen found in the filtrate from the coagulum. This last may be obtained by evaporating the alcohol solution of the flour to small volume, adding water, and then bringing nearly to the boil. When this process has been repeated two or three times, the coagulum is filtered off. H. F. E. H.

Analysis of Crude Glycerol by the International Standard Methods, 1911. Estimation of Organic Residue. R. G. Grimwood. (*J. Soc. Chem. Ind.*, 1913, **32**, 1039-1041.)—The author describes processes which shorten the time which is required for the initial evaporation of the glycerol in the estimation of the organic residue in crude glycerols (*cf.* ANALYST, 1911, **36**, 315); a description of an electrically heated oven is also given. Two methods of carrying away the glycerol vapour have been found to be effective. In the first a small electric fan is so arranged that a current of air is drawn into the oven and propelled over the surface of the contents of the dish; in the second method air under slight pressure is supplied by an ordinary water-blower and conveyed through suitable tubing to the oven, where it is distributed through small jets in the sides of copper tubing running round the internal walls of the oven. The oven consists of an outer uralite shell, between which and the inner shell of sheet iron, or oven proper, coils of special resistance wire are fixed along the bottom and two of the sides. The door consists of a metal plate with an outer covering of uralite. Perforated sheet iron shelves, about 4 inches wide, run from front to back at each side of the oven, leaving a clear open space in the centre of the oven from top to bottom and from front to back. The shelves are provided with levelling screws, and the tubes for the air-supply are arranged at a suitable height above the shelves. Openings are provided in the top of the oven for the escape of the glycerol vapours, and for the thermometer. The door is fitted with a covered port-hole for inspection, and an electric-light bulb is fixed inside the oven. On a 100-volt alternating circuit the oven takes 9·2 amps. to raise the temperature to 156° C. in fifty minutes; a resistance is then inserted, reducing the current to 8·4 amps., when the temperature rises to 160° C. in ten minutes and remains constant. The variation of temperature at any part of the interior of the oven does not exceed 2·2° C.

Estimation of Organic Residue at 160° C.—The quantities having been prepared in accordance with the method prescribed by the International Standard Method (*loc. cit.*), the dishes are placed on the top of the oven and the excess of water is evaporated. The temperature of the oven having been adjusted at 150° to 160° C., the air supply from the blower is turned on, the dishes are placed on the two top shelves, and the door is closed quickly. At the end of thirty minutes, and of each subsequent thirty minutes, the condition of the residues is observed through the port-hole. If the glycerol in any of the dishes has skinned over, the dish is removed, a few drops of water added, the crust broken up, and the dish returned to the oven. Should a pool form owing to inequalities on the surface of the dish, the glycerol is redistributed. Most crude glycerols at the end of one hour, and most saponification glycerols at the end of two hours, will have evaporated sufficiently to permit of their treatment for the first baking, which is then carried out exactly as prescribed. Thin Jena-glass dishes, 80 mm. in diameter and 12 mm. in depth, weighing from 15 to 20 grms., are recommended for use in the estimation.

Acetylation of Residues.—The evaporation of the water extract of a residue in the acetylating flask may be carried out expeditiously by attaching the flask to a water-cooled condenser fixed in a filter-pump flask and connected with an exhaust pump. On heating the flask in a water-bath to from 70° to 80° C., the water distils rapidly, leaving the residue ready for acetylation. Four or five residue extracts may be prepared for acetylation within one hour. W. P. S.

Estimation of Volatile Oils in Aromatic Plants, Drugs, and Spices by the Tailameter. P. C. Chattopadhyay. (*J. Soc. Chem. Ind.*, 1913, **32**, 968.)—The sample is distilled with steam into the apparatus, which consists of a flask with a long neck graduated in tenths of a c.c. for 10 c.c. A swan neck, closed with a stopcock, is sealed into the flask near the bottom. Surplus distillate is run off through the swan neck, and when the distillation is finished the oil is brought within the graduations of the neck by adding or removing distillate. If 100 grms. are taken for analysis, the volume observed is the percentage of oil to one place of decimals; if 1,000 grms., to two places, and so on. In general the solubility of the oil in water is negligible; if not, the solubility is determined and allowance made for it.

O. E. M.

Constituents of Senna Leaves. F. Tutin. (*J. Chem. Soc.*, 1913, **104**, 2006-2023.)—The material employed consisted of (1) Tinnevelly senna leaves (*Cassia angustifolia*, Vahl); (2) senna leaves from Lima, Peru, botanically identical with (1); (3) Alexandrian senna leaves (possibly *Cassia acutifolia*). (1) An alcoholic extract of Tinnevelly leaves when distilled with steam yielded a small amount of essential oil. From the portion of the extract which was soluble in water the following substances were isolated: (i.) Salicylic acid; (ii.) rhein, $C_{15}H_8O_6$; (iii.) kaempferol, $C_{15}H_{10}O_6$; (iv.) aloe-emodin, $C_{15}H_{10}O_5$; (v.) kaempferin, $C_{27}H_{30}O_{16}6H_2O$ (m.pt., 185° to 195° C.), a new glucoside of kaempferol; (vi.) a mixture of the glucosides of rhein and aloe-emodin; (vii.) the magnesium salt of an unidentified organic acid. The aqueous liquid also contained a quantity of a sugar yielding *d*-phenylglucosazone and some brown amorphous products, which, on treatment with alkali, gave kaempferol and small quantities of rhein and aloe-emodin. The portion of the alcoholic extract insoluble in water consisted of a soft, dark green resin amounting to 7 per cent. of the weight of leaves used. From this were isolated chlorophyll, myricyl alcohol, a phytosterol ($C_{27}H_{46}O$), a phytosterolin ($C_{33}H_{56}O_6$), and palmitic and stearic acids in addition to some of the substances mentioned above. (2) The senna leaves from Lima, Peru, contained all the above-mentioned compounds except the magnesium salt, and, in addition, isorhamnetin. A glucoside of isorhamnetin mixed with glucosides of kaempferol, rhein, and aloe-emodin was also present. (3) Alexandrian leaves yielded in addition to myricyl alcohol and a phytosterolin, rhein, aloe-emodin, kaempferol, and isorhamnetin. These four substances were also present in the form of glucosides, and in much greater proportion than in the free state. The author cannot confirm the statements of Tschirch and Hiepe (*Arch. Pharm.*, 1900, **238**, 427) that senna leaves contain "sennaisoemodin," "sennachrysophanic acid" (chrysophanol), and a "substance $C_{14}H_{10}O_5$," as he ascertained that the anthraquinone derivatives present consist solely of rhein and aloe-emodin, equal quantities of which if mixed would, it is true, have the empirical composition and properties assigned by Tschirch and Hiepe to the "substance $C_{14}H_{10}O_6$." The "sennarhamnetin" of these authors has been found to be identical with the isorhamnetin described by Perkin (*J. Chem. Soc.*, 1896, **69**, 1658). H. F. E. H.

Rapid Estimation of Water in Foods and Feeding Stuffs. A. Fornet. (*Chem. Zeit.*, 1913, **37**, 1400.)—For the estimation of water in flour, etc., heating for four hours at 105° C. in a circulating current of air gives accurate results. For practical purposes a rapid estimation may be made by heating the sample for ten minutes at temperatures of 160° to 180° C. The results thus obtained, even in the case of easily decomposable substances, such as fat and albumin, agree well with those obtained by long-continued heating at lower temperatures in a drying oven. The author has devised a combination of heating oven and balance, with which two samples may be heated at the same time at exactly 160° C., and the percentage of water read directly upon a scale. In the case of starch the results are somewhat higher and more accurate if the sample be given a preliminary drying at 50° C. In every instance, however, the time of heating should be based upon the results of exact estimations by older methods of the moisture in substances of the same kind.

C. A. M.

Composition of Cider Vinegars made by Generator Process. L. M. Tolman and E. H. Goodnow. (*J. Ind. and Eng. Chem.*, 1913, **5**, 928-933.)—Most of the figures for cider vinegar to be found in the literature refer to samples manufactured by the old barrel process, which was so little under control that widely divergent products, such as the analyses disclose, were to be expected. The modern generator process, however, might be expected to yield products more uniform in character, and, so far as differences exist, they should be traceable to differences in the cider used and be nearly independent of the small, uncontrollable variations of the process. A few analyses of alleged genuine cider vinegars made by the generator process have been published, but the authenticity of the samples was not beyond question, and any figures based on less than, say, two months' continuous testing are apt to be misleading. A brief description of the process will make this clear. In the factory in which the present research was carried out, a large number of comparatively small generators are used, but the ratio of generator contents to daily production is nearly constant for this type of plant. Each generator holds 200 gallons of vinegar; only 40 gallons of vinegar drips from each daily, and the feed consists not of 40 gallons of cider but of a mixture of 18 gallons of cider with 22 gallons of vinegar from the previous day's drip. Only by operating on a large bulk of cider of uniform composition and making observations over a prolonged period can numbers be obtained from which it is safe to draw any general conclusions. The authors' observations extended over two months, during which some 40,000 gallons of cider were converted into vinegar, of which the bulk was so near 40,000 gallons that no correction is necessary before instituting a comparison between the cider and resultant vinegar.

The cider contained on the average 7·7 per cent. by volume of alcohol and 0·27 grm. acetic acid per 100 c.c., the vinegar 5·77 grms. acetic acid per 100 c.c. This is only 70 per cent. of the theoretical yield, but allowing for the alcohol in the finished vinegar (about 0·4 per cent.), the loss of alcohol is only a little over 20 per cent. These figures are more likely to vary between one plant and another than those which follow.

It is shown that the total solids, ash, and glycerol content are so little affected by the process that figures which can be accepted as limits for genuine cider may be applied equally to cider vinegars. The non-sugars appear to decrease slightly and the sugars to increase, if the reducing action on Fehling's solution after inversion be assumed to measure nothing but sugar; but it is shown that the process gives rise to the production of substances of aldehydic nature—not acetaldehyde—which may cause the sugars to be overestimated by 0·15 to 0·2 grm. per 100 c.c. unless the vinegar be repeatedly evaporated to expel them. When this precaution is taken, it is found that the proportion of non-sugars calculated from the difference between the total solids and true sugar content is substantially identical for the vinegar and the cider from which it is made.

Apart from the acetification of the alcohol, the most important difference between cider and cider vinegar lies in the content of fixed acids. This is diminished enormously, as in the barrel process of acetification, and as in the alcoholic fermentation which precedes either process, and a figure as low as 0·04 per cent. (calculated as malic acid) must not be taken as evidence that an alleged cider vinegar is not genuine. In the works where this research was carried out, the average was about 0·06 per cent., and 0·04 per cent. is not necessarily the lowest limit for genuine cider vinegar, though the lowest figure recorded in the paper. On the other hand, genuine cider vinegar should always contain pentosans, the amount of which appears always to increase during the process, and may increase 50 per cent. G. C. J.

Estimation of Lactic Acid in Wine. T. Roettgen. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1913, **26**, 437-439.)—Results of experiments are recorded showing that the total solids of a wine contain the whole of the lactic acid present in the latter, the same amount of the acid being found whether the estimation is made on the wine direct or on the total solids; only very small amounts (about 0·005 grm. per 100 c.c. of wine) of volatile acids are retained in the total solids. The methods proposed by Möslinger and Kunz for the estimation of lactic acid were found to be equally trustworthy. W. P. S.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Analyses of Ascitic Fluid. F. Raynaud. (*J. Pharm. Chim.*, 1913, **8**, 458-460.)—The author has examined the ascitic fluid drawn from a man within a period of three weeks; five tappings yield a total volume of 65 litres of liquid. The fluid was turbid, neutral to litmus, acid to phenolphthaleïn, and had a sp. gr. varying from 1·008 to 1·011. It contained—total solids, 15·7 to 21·2; ash, 7·6 to 8·1; chlorides (as sodium chloride), 6·51 to 6·45; alkali-albumin, 0·23 to 0·67; globulin, 3·8 to 4·9; serin, 1·65 to 2·6 grms. per litre. Phosphates and lecithin were present in traces, but urea, sugars, fats, cholesterol, blood, biliary pigments, indoxyl, urobilin, fibrin, aceto-albumin, albumose, and peptone were absent. Rivalta's test yielded a negative reaction. W. P. S.

Estimation of Acidity in Meal, Grist, and Bread, taking into account the Action of Bacteria and Enzymes. O. Rammstedt. (*Zeitsch. angew. Chem.*, 1913, **26**, 677-680.)—Of a large number of methods proposed for the estimation of acidity in cereal meals, some are based on extraction with alcohol, others on extraction with water. The method of Kreis and Arragon is adopted in Switzerland: 10 grms. of meal are digested with 100 c.c. of water for thirty minutes on a boiling-water bath; 0·5 c.c. of a 2 per cent. solution of phenolphthaleïn is added, and the whole titrated with $\frac{N}{10}$ sodium hydroxide. This process is open to the objection that enzymes and bacteria begin to be active as soon as the sample is mixed with water, thereby considerably increasing the acidity. Moreover, the change in colour of the indicator is obscured by the presence of the meal, particularly in the case of yellow maize, and this also tends to give high results. In order to eliminate the action of enzymes, alcoholic extraction must be employed, and the author recommends the following modification of Schindler's method: 10 grms. of the very finely ground sample, sifted through a No. 6 miller's gauze, are treated with 100 c.c. of boiling, neutral, and absolute alcohol. The mixture is boiled under a reflux condenser for thirty minutes on the water-bath, then filtered, and the residue washed three times with 10 c.c. of neutral absolute alcohol. The filtrate and washings are titrated with $\frac{N}{10}$ sodium hydroxide in presence of phenolphthaleïn. The acidity is expressed in terms of c.c. of $\frac{N}{1}$-alkali per 100 grms. of the sample. The fine grinding of the sample is of the utmost importance. For the estimation of acid in bread, 20 grms. of the crumb portion are treated in an Erlenmeyer flask with 175 c.c. of boiling alcohol in such a way that at first only sufficient alcohol is added to moisten the bread; the lumps are broken down with a rod, and the remainder of the alcohol is added gradually so that a uniform suspension is obtained. After boiling for thirty minutes as described above, the extract is filtered off, and the residue washed three times with 25 c.c. of alcohol. The total filtrate is titrated as before. Working with aqueous extraction, the author has been able to show that there is a separate action both of bacteria and enzymes in increasing the acidity. If the estimation of the pre-existing acidity of the sample is required, only the alcohol method is admissible; but if it be desired to obtain an idea of the activity of the enzymes or the behaviour of the meal on fermentation, the Kreis-Arragon method may be used in conjunction with the alcohol method, and conclusions drawn from the difference between the results. J. F. B.

New Method for the Estimation of Urea in Blood. E. K. Marshall, jun. (*J. Biol. Chem.*, 1913, **15**, 497-494; through *Chem. Zentralbl.*, 1913, II., 1827.)—The author's method is applicable for the estimation of urea in blood. The urea in 10 c.c. of defibrinated serum is converted into ammonium carbonate by treatment with soya-bean extract, and, after acidifying with mineral acid, the ammonia is estimated by Folin's method, being driven over into a measured volume of $\frac{N}{25}$ hydrochloric acid by means of a current of air. The ammonia originally present in the blood is estimated in a separate experiment, and the nitrogen of the urea is found by difference. The method may be employed without the previous removal of the proteins. It is also applicable to other body-fluids, in which case the urea is first extracted from the tissues with alcohol, the extract evaporated, and the residue dissolved in water. Strongly acid liquids and liquids containing mercuric chloride are unsuitable.

J. F. B.

Guaiacum Resin as a Reagent for the Detection of Oxydases and of Minute Traces of Copper. W. R. G. Atkins. (*Chem. Soc. Proc.*, 1913, **29**, 303.)—In order to ascertain how far traces of metals might vitiate tests for oxydases by guaiacum resin, a series of experiments was carried out to determine the limits of sensitiveness of the reaction towards certain salts. Adopting the methods usual in water analysis, it was found that very minute amounts of copper salts or of potassium permanganate may be detected by this test. Accordingly it is brought forward as a reaction of utility in water analysis. Below are recorded the limits of sensitiveness of the reaction, expressed in grms. per c.c. Copper as sulphate, 2×10^{-9} to 2×10^{-8}; potassium permanganate, 4×10^{-8}; potassium dichromate, 1×10^{-7}; iron, as ferrous sulphate, 1×10^{-6}, as ferric sulphate, 1×10^{-6}; lead as acetate, 6×10^{-6}; nitric acid, 2×10^{-5}; manganese as sulphate, 8×10^{-4}. In each case a few drops of hydrogen peroxide were added, as well as a very dilute solution of the reagent. Traces of chlorides were also present, and play an important part.

Indigo-Forming Substances in Urine. R. V. Stanford. (*Zeitsch. Physiol. Chem.*, 1913, **88**, 47-56).)—The numerous oxidation processes that have been described were found to be unsatisfactory, owing mainly to the danger of over-oxidation. In many cases decolorisation is caused by the oxidising action of chlorine compounds present as impurities in the chloroform used; access of air has a similar effect. The author finds that if pure chloroform is used, and the oxidation be carried out with hydrogen peroxide and hydrochloric acid in a current of carbon dioxide, perfectly concordant results can be obtained. The chloroform solution is pure blue, and the indigo can be estimated approximately by comparing an aliquot portion colorimetrically with Fehling's solution. E. W.

Centrifugal Method for Estimating Proteins in Urine. C. Strzyzowski. (*Zeitsch. Physiol. Chem.*, 1913, **88**, 25-38.)—The author finds that the proteins in urine can be rapidly and accurately estimated by measuring the volume of precipitate formed after centrifuging. The density and temperature of the liquids used, the time between precipitation and centrifuging, the number of revolutions per minute, and the duration of centrifuging, must be identical for all estimations. The density of the urine is made up to 1·030 by the addition of salt, and to 5 c.c. of this urine 2 c.c. of a zinc sulphate solution (sp. gr. 1·300, prepared by dissolving 112·3 grms. zinc sulphate in 100 c.c. water and 3 c.c. glacial acetic acid, and making up to 200 c.c. at 15°) are added, and the mixture shaken. Three c.c. of Esbach's solution (1 grm. picric acid + 2 grms. citric acid made up to 100 c.c.) are then allowed to flow slowly on the surface of the mixed liquids, the mixture shaken five times, and, after one minute, centrifuged during fifteen minutes at 2,000 revolutions per minute. Tables are given showing volumes of precipitate and corresponding protein content for a number of different temperatures. E. W.

Sugars in Urine in Cases of Pentosuria. E. Zerner and R. Waltuch. (*Monatsh.*, 1913, **34**, 1639-1652.)—It is commonly accepted, on the authority of Neuberg (*Ber.*, 1900, **33**, 2243), that in cases of pentosuria the characteristic sugar in the urine is *d-l*-arabinose. Elliot and Raper, however (*J. Biol. Chem.*, 1912, **11**, 213), were unable to isolate this sugar, and concluded that the pentose in such urines was probably *d-l*-ribose. A sample of urine examined by the authors had a specific gravity of 1·025, gave the usual pentose reactions, and contained 0·34 per cent. of sugar (as dextrose by Allihn's method). By the furfural method it yielded phloroglucide corresponding to 0·135 per cent. of arabinose—a result which rendered the presence of xyloketose improbable. An osazone melting at 160° to 161° C., and having $a_D = +0{\cdot}71°$, was separated from the urine, and when this was mixed with *l*-xylosazone (m.pt. 162° to 163° C.), a mixture was obtained melting at 208° to 210° C., which agreed well with the melting-point of Fischer's *d-l*-xylosazone (210° to 215° C.). From this the authors concluded that the pentose in the urine was *d*-xylose. The fact that the concentrated urine was optically lævorotatory rendered the presence of *l*-xylose improbable. Similar results were obtained with the urine in another case of pentosuria. C. A. M.

ORGANIC ANALYSIS.

Beeswax of the Viking Period. J. Sebelien. (*Zeits. angew. Chem.*, 1913, **26**, 689-692.)—Tablets of beeswax found in a ship buried in the grave of a Viking queen (*circa* A.D. 800) at Oseberg in Norway gave the following analytical values: Sp. gr. at 15° C., 0·962; m.pt., 63° C.; acid value, 16·6; ester value, 78·74; ratio of acid value to ester value, 4·74; and iodine value, 6·02. The composition of the wax thus fell within the limits recorded for normal beeswax, although the ratio value was higher than that usually found in the case of modern Norwegian wax (4·07 to 4·26). The vegetable débris (separated by treating the wax with xylene) included fragments of conifer wood, barley and oat starch granules, and pollen grains, which were identified as having belonged to plants of the same families as those visited by honey bees in Norway at the present day. C. A. M.

Action of Calcium on Acetone. P. N. Raikow. (*Chem. Zeit.*, 1913, **37**, 1455.)—When anhydrous acetone is left in contact with metallic calcium shavings, evolution of hydrogen takes place after some time, and in the course of about ten days the liquid becomes golden-yellow. When distilled, it yields a colourless distillate with a fruit-like odour, and consisting in the main of mesityloxide $CH_3.CO.CH:C(CH_3)_2$. C. A. M.

Folin Method for the Estimation of Creatine and Creatinine. W. H. Thompson, T. A. Wallace, and H. G. S. Clotworthy. (*Biochem. J.*, 1913, **7**, 445-466.)—For control of the bichromate standard solution, creatinine picrate, or the double picrate of creatinine and potassium, is recommended. For the estimation of creatine in weak solutions, the best results were obtained by using an equal quantity of $\frac{N}{1}$ hydrochloric acid and heating either on the water-bath for three hours or in the autoclave for twenty-five minutes at 117° C. The optimum time and temperature for the development of the colour was seven minutes at 15° to 17° C. The addition of too much alkali reduces the colour; for aqueous solutions and urine the best results are given by the quantity recommended by Folin—viz., 5 c.c. of 10 per cent. caustic soda in excess of that required to neutralise. For accurate work the readings on the colorimeter scale can only be regarded as strictly proportional if they lie between a lower limit of 7 mm. and an upper one of 9 mm. If separated by a wider interval, a correction factor has to be applied. The darkening of the pigments of urine which occurs on boiling with $\frac{N}{1}$ hydrochloric acid, adds to the estimate of creatine and creatinine contained in it. With human urine, the increase is slight (1 to 2½ per cent.), with dog's urine as much as 10 per cent. Dextrose to the extent of 10 per cent. does not affect the estimation of creatine, and phosphoric acid had no advantages as compared with hydrochloric acid in estimating creatine when dextrose is present. The recovery of added creatine from diabetic urine gave results lower by 5 per cent. than from aqueous or sugar solutions. The smaller recovery is probably due to the effect of aceto-acetic acid. Identical results were obtained whether a water-bath or autoclave was used in the estimation. E. W.

Estimation of Hydroxy Fatty Acids in Fats by Means of Magnesium Methyl Iodide. T. Zerewitinoff. (*Zeitsch. anal. Chem.*, 1913, **51**, 729-737.)—The method depends on the reaction taking place between magnesium methyl iodide and substances containing hydroxyl groups, methane being liberated according to the equation:

$$CH_3MgI + R.OH = CH_4 + R.OMgI.$$

This reaction has been described previously by the author (ANALYST, 1907, **32**, 299; 1908, **33**, 366; 1911, **36**, 117; 1912, **37**, 571.)—In the case of fatty acids, the carboxyl (- COOH) groups also react; for instance, stearic acid yields one hydroxyl group; ricinoleic acid, two groups; and dihydroxy-stearic acid, three groups. In each of these cases one of the hydroxyl groups is derived from the carboxyl group. The carboxyl-hydroxyl groups are first estimated by titrating a quantity of the fatty acid with alcoholic potassium hydroxide solution; the following formula gives the carboxyl value:

$$\text{OH\%} = \frac{17 \times T \times n \times 100}{56 \times a} = 30{\cdot}357\frac{T \times n}{a}$$

where 17 = molecular weight of hydroxyl, 56 = molecular weight of potassium hydroxide, T = the titre of the alkali solution, n = the number of c.c. of this solution used for the titration, and a = the weight of the fatty acid in grms.

The total hydroxyl value is then estimated by means of the process and apparatus described previously: About 0·2 grm. of the fatty acid is used for the estimation; this is dissolved in 15 c.c. of pyridine and treated in the apparatus with magnesium methyl iodide dissolved in amylic ether. The total hydroxyl value is calculated from the formula:

$$\text{OH\%} = \frac{0{\cdot}000719 \times V \times 17 \times 100}{16 \times S} = 0{\cdot}0764\frac{V}{S},$$

where 0·000719 = the weight of 1 c.c. of methane at 0° C. and 760 mm. pressure; 16 = the molecular weight of methane; 17 = the molecular weight of hydroxyl; V = the volume (in c.c.) of methane at 0° C. and 760 mm.; and S = weight of fatty acid taken. The difference between the two estimations gives the hydroxyl value of the fatty acid. Results of estimations of the hydroxyl value of various fatty acids are given, showing that the method yields results which agree with those found by the acetyl method.

W. P. S.

Estimation of Nicotine in the Presence of Ammonia. R. Spallino.

(*Gazz. Chim. Ital.*, 1913, **43**, 493-500.)—The method depends upon the fact that in alcoholic solution nicotine behaves towards picric acid as a mono-acid base, whereas in aqueous solution it forms a di-picrate. Hence if a mixture of nicotine and ammonia be titrated with a standard solution of picric acid first in aqueous and then in alcoholic solution, three molecules of the acid will be consumed in the first titration as against two molecules in the second, and the amount of nicotine may be calculated from the difference between the results. In practice it is better to add an excess of picric acid and to titrate the uncombined acid. For example, in estimating the nicotine in tobacco, from 1·5 to 3·5 grms. of the sample are distilled over magnesia in a current of steam, and the distillate received in 50 to 100 c.c. of a solution of picric acid (1 c.c. = about 0·01 grm.). After 600 to 700 c.c. have been collected, the distillate is made up to a litre with alcohol, warmed until a clear solution is obtained, and divided into two equal parts, which are evaporated to dryness. One of the residues is dissolved in alcohol and the solution titrated with $\frac{N}{20}$ baryta solution with lacmoid as indicator. The other residue is taken up with water, the solution made up to 100 c.c. and filtered, and 75 c.c. of the filtrate titrated as before. The difference between the amounts of uncombined picric acid corresponds to the quantity of nicotine present. The results are shown to be concordant and accurate.

C. A. M.

Estimation of Nicotine as Silicotungstate. R. Spallino. (*Gazz. Chim. Ital.*, 1913, **43**, 482-486.)—Good results are obtained by this method when the silicotungstate is ignited and the residue, $WO_3 + SiO_2$, weighed. Chapin's method of weighing the precipitate after drying at 125° C. (ANALYST, 1911, **36**, 544) is inaccurate, since constant results cannot be obtained. Eventually, if the heating at 125° C. be continued for thirty-four hours, a residue of $WO_3 + SiO_2$ is left. At a lower temperature (115° C.) sufficient water is not expelled to leave the silicotungstate, $12WO_3,SiO_2,2H_2O.3C_{10}H_{14}N_2$, described by Chapin (*loc. cit.*). C. A. M.

Barium and Arsenic in Italian Tobacco. R. Spallino. (*Gazz. Chim. Ital.*, 1913, **43**, 475-481.)—Ten representative samples of Italian tobacco prepared for smoking or for snuff contained from 0·019 to 0·036 per cent. of barium (calculated as sulphate). Part of the barium (0·003 to 0·009 per cent. of the dry tobacco) was soluble in water. Arsenic was also found in all the samples examined, in quantities ranging from 0·05 mgm. to 0·51 mgm. per 50 grms. of the dry tobacco. The tobacco ash was free from arsenic, except in the case of the sample containing 0·51 mgm., the ash of which showed a faint trace. In view of the common use of arsenical insecticides, estimation of the arsenic in tobacco leaves from special districts would be advisable. C. A. M.

Titanium and Rare Metals in Tobacco Ash. F. T. Mosca. (*Gazz. Chim. Ital.*, 1913, **43**, 437-440.)—In the ash of leaves of Kentucky tobacco grown in Italy, the author detected barium, cæsium, lithium, and titanium. One grm. of the ash, when fused with potassium bisulphate and dissolved in water acidulated with sulphuric acid, gave a solution in which titanium could be identified by Schönn's hydrogen peroxide reaction, and by the thymol reaction of Lenher and Crawford (ANALYST, 1912, **37**, 590). Its presence was also confirmed by the spectroscope.

C. A. M.

Shrewsbury's Reaction for Paraffin. P. A. Meerburg. (*Chem. Weekbl.*, 1913, **10**, 742; through *Chem. Zentralbl.*, 1913, **11**, 1778.)—In testing fats for paraffin by the method of Shrewsbury (ANALYST, 1909, **34**, 348), the saponified mass should be added to the methyl alcohol at once, instead of drop by drop. To dissolve the soap, the mixture must be warmed and then cooled. If the saponification is carried out in a Jena flask, the glass is attacked by the solution of sodium hydroxide in glycerol, and the solution becomes turbid. A metal vessel is thus better for this purpose. Even then quantities of paraffin below 0·1 per cent. cannot be detected with certainty. O. E. M.

Analysis of Petroleum Oil and Paraffin Wax. H. Kantorowicz. (*Chem. Zeit.*, 1913, **37**, 1394-1396; 1438-1441.)

Estimation of Asphaltum.—The alcohol-ether method of Holde and Meyerheim, and Holde's "normal benzine" method do not yield a pure asphaltum, owing to the disturbing influence of paraffin wax, ceresin, and petroleum oil upon the action of the solvents. Hot ethyl acetate, however, readily dissolves the oil, paraffin wax, and ceresin, leaving a residue of asphaltum. Five grms. of the oil are boiled for fifteen minutes beneath a reflux condenser with 220 c.c. of ethyl acetate, and the solution filtered while hot. The residue in the flask is boiled with successive portions of 20 c.c. of the solvent until the washings are colourless, and the contents of the filter also washed in the same way. The asphaltum is then washed with hot benzene into a tared glass basin, the solution evaporated, and the residue once more washed, if necessary, with 50 c.c. of boiling ethyl acetate, and dried and weighed. The following comparative results were thus obtained :

Cylinder Oil.	Benzine Asphaltum. Per Cent.	Ethyl Acetate Asphaltum. Per Cent.
Roumanian	0·15–0·20	0·0–0·05
German	0·26	0·06
	0·20	0·02
	0	0
American	0	0·06
	0	0·08
	0	0·27
	0·47	0·61
	0	0
Russian	0·79 ; 0·54	0·04

The residues obtained by the alcohol-ether method were invariably higher, but this was found to be due to their consisting of a mixture of hard asphaltum and ceresin. These results show that the absence of asphaltum cannot be definitely asserted from a negative result in the benzene test.

Estimation of Paraffin Wax and Ceresin.—Processes in which preliminary distillation is used are unsuitable, since ceresins are decomposed by the heat. The method of precipitating the wax at a low temperature with a mixture of alcohol and ether is also frequently inapplicable, for many oils are insoluble in the mixture even at +15° C. Petroleum spirit and "normal benzine" give widely varying results with the same paraffin wax at −20° C., whilst ethyl acetate varies in its solvent action upon oils of different origin. Good results are obtained, however, by the use of propyl acetate, in which heavy machine oils will remain in solution even at −20° C. Five grms. of petroleum residue are extracted with ethyl acetate as described above, and the filtrate and washings distilled. The residue is heated on a brine bath in a current of air to expel the last traces of ethyl acetate, and is then dissolved in 40 c.c. of propyl acetate, with the aid of heat if necessary. The solution is cooled to −20° C., and filtered at that temperature in Holde's apparatus, the precipitate washed with 80 c.c. of propyl acetate at −20° C., and dissolved in hot benzene, the solution evaporated, and the residue of paraffin wax and ceresin dried and weighed. The wax is free from oil, and, except for being of a dark colour, is pure. The results are higher than those given by precipitation with alcohol and ether, this being partly due to the omission of the preliminary distillation. C. A. M.

Some Observations on the Abel Heat Test. B. J. Smart. (*J. Soc. Chem. Ind.*, 1913, **32**, 967.)—In reply to Egerton (Analyst, 1913, **38**, 275), who on some points differs from Robertson and Smart (ANALYST, 1910, **35**, 276) the author points out that if the oxidation of potassium iodide on the test paper is slow enough, no free iodine will appear, as it will all be converted into hypoiodite, and the test will be of indefinite duration. The higher value of K obtained by Egerton in the equation $TC = K$ is due to depletion of the nitric peroxide in the comparatively small vessels used by him, and to the reactivity of their relative large glass walls. If "easily removable nitric peroxide" were absorbed as nitric acid by guncotton, it would be removable with difficulty, and would accelerate the decomposition even in the Will test; the water taken up by guncotton is constitutional. The behaviour of the heat test paper is not directly comparable with that of the naphthylamine drop, which depends upon the diazo reaction, and consequently solely on nitrous acid. The fall in rate of evolution of nitric acid shown by Egerton's continuous tests may be due to reduction of nitric peroxide to nitrous acid by small quantities of adherent material. This may hold to a much smaller extent with the paper test, but drying of the paper plays an important part. The difference in their physical state explains the difference in the behaviour of guncotton fibre and of nitroglycerine comminuted on quartz wool in absorbing or evolving nitric peroxide; the contradiction is only apparent. The comparison between the heat test and the dissociation of copper sulphate does not hold; apart from catalysis, guncotton could be completely decomposed, while with copper sulphate the reaction is reversible, and ceases when solution-pressure is reached. The author emphasises the importance of taking the physical condition of the explosive subjected to the Abel test into consideration. O. E. M.

Estimation of Sulphuric Acid in Nitrocelluloses. Piest. (*Zeitsch. angew. Chem.*, 1913, **26**, 661-663.)—The author describes seven methods which have been recommended for this purpose. Of these methods, five are said to be worthless, and another to be frequently attended with mechanical loss due to miniature explosions. The following method is recommended: The nitrocellulose (20 grms.) is decomposed by treatment with 70 c.c. nitric acid and 1 grm. potassium nitrate, the mixture being evaporated to dryness on the water-bath. The residue is taken up in dilute hydrochloric acid, the solution filtered, and sulphuric acid precipitated as barium sulphate. Cotton intended for the manufacture of nitrocellulose may be examined in a similar manner, 10 grms. being added in small portions at a time to 50 c.c. of nitric acid containing 0·5 grm. potassium nitrate. The author's experiments do not support the view that the sulphuric acid found in nitrocelluloses is present as a mixed ester, nor that there is any necessary connection between stability and sulphuric acid content. If mixed esters are formed, they would be decomposed in the washing process. On the other hand, the acid so liberated might require exceptionally prolonged washing to remove it, and a sulphuric acid content much above the normal (0·02-0·06 per cent.) may indicate insufficient washing, but may be due to nothing more than the use of a gypseous water for this purpose. G. C. J.

Determination of a Small Amount of Reducing Sugars in the Presence of a Large Proportion of Cane Sugar. H. Pellet. (*Bull. Assoc. Chim. Sucr. Dist.*, 1913, **31**, 183-205.)—When the usual method of determining a small amount of reducing sugar in the presence of a large proportion of cane sugar by means of Fehling's solution is applied, the result cannot be strictly accurate owing to the hydrolysis of cane sugar by the action of alkali during boiling, the error being quite an appreciable one in the examination of factory white or refined sugars. It has been proposed to use a Fehling's solution in which the sodium hydroxide has been replaced by sodium or potassium carbonate or bicarbonate, but it is now stated that with these reagents cane sugar always causes a slight reduction, and that for dealing with small amounts of reducing sugars the sensitiveness is insufficient. Satisfactory results are claimed to be obtainable by the author's new procedure, in which a Fehling's solution containing the minimum amount of sodium hydroxide is used, and heating is carried on at a comparatively low temperature. As usual, the Fehling's solution is prepared in two separate portions: (*a*) has 34·64 grms. of pure cupric sulphate per 500 c.c.; while (*b*) contains 180 grms. of Rochelle salt and 60 grms. of sodium hydroxide in 500 c.c. of water; equal volumes of the solutions (*a*) and (*b*) being mixed previous to use to give a liquid 10 c.c. of which = 0·05 grm. of invert sugar. For standardising this copper solution, a pure solution of invert sugar, 1 c.c. of which = 0·005 grm., is prepared as follows: 4·75 grms. of pure cane sugar are dissolved in about 250 c.c. of water contained in a litre flask, 2 c.c. of concentrated hydrochloric acid added, and hydrolysis effected by placing the flask in a boiling-water bath for thirty minutes. After adding 0·5 grm. of salicylic acid, and neutralising with sodium hydroxide, the liquid is cooled, and made up to the mark.

Procedure.—Twenty c.c. of the Fehling's solution, 20 c.c. of a solution of the sample (containing less than 0·1 grm. of reducing sugars), and 10 c.c. of water, are placed in a beaker, and heated in a water-bath at 60° to 62° C. for exactly ten minutes. The resulting cuprous oxide may be determined by the Herzfeld or other convenient method, but the following process of the author is recommended, being both accurate and expeditious: Immediately after removal from the water-bath, the assay liquid is diluted with 50 c.c. of cold water, filtered twice through a previously moistened small paper, and the precipitate washed well with warm water. After the incineration of the filter in the muffle (not over a gas flame), the resulting cupric oxide is weighed, and the amount due to the copper fixed by the filter-paper, as ascertained by a blank determination, is deducted. On multiplying the difference by the factor established with the standard invert sugar solution, usually 0·453, the amount of reducing sugars in the sample is found. For special work a colorimetric scale may be prepared by passing the cuprous oxide obtained from different known amounts of the pure invert sugar solution through discs of paper of a uniform size—say, 3 cm. in diameter—and carrying out the actual determination under identical conditions, these colour discs being used as standards. J. P. O.

Orthotolidine as Reagent for Colorimetric Estimation of Small Quantities of Free Chlorine. J. W. Ellms and S. J. Hauser. (*J. Ind. and Eng. Chem.*, 1913, **5**, 914-916.)—E. B. Phelps suggested the use of an acetic acid solution of *o*-tolidine for the detection of such small quantities of chlorine as may be present in drinking water which has been treated with hypochlorite. According to him, a yellow colour is developed in presence of 0·05 part of chlorine per million. Dittoe and Van Buskirk (*Ohio State Board of Health*, 1913, *Bull.* 3) made use of the method, but more often obtained a green colour, and in one instance a blue. The original method is therefore not adapted to quantitative work. Moreover, the colour is evanescent, not persisting for more than five minutes with small amounts of chlorine, and the acetic acid solution of the reagent darkens with age.

It is now found that the variable results obtained by Dittoe and Van Buskirk are dependent on the hydrion concentration. It is waters originally alkaline that develop a blue colour, and a high concentration of acetic acid is necessary to reduce this to a yellowish-green. All Dittoe and Van Buskirk's solutions were "acid." In presence of a very little free mineral acid, a yellow colour (red in high concentrations of chlorine) is produced, which is not affected by further additions of acid, is permanent for at least thirty minutes, and intense enough to make possible the detection of 0·005 part of free chlorine per million. It is also found that a hydrochloric acid solution of the reagent does not deteriorate on storage. A convenient strength is 1 grm. of *o*-tolidine in 1,000 c.c. of 10 per cent. hydrochloric acid, and 1 c.c. of this solution is added to 100 c.c. of the water to be tested. Quantitative comparisons may be made with standards up to 10 parts per million, but with concentrations above 3 parts per million more than 1 c.c. of the reagent should be used. The preparation of standards for comparison with very dilute solutions requires the use of exceptionally pure distilled water if the standard is to be a chlorine solution of known strength. For this reason the use of permanent standards may prove more convenient. The paler tints may be matched by a very dilute solution of bichromate, but to match the deeper ones a blue element must be added. The authors make use of copper sulphate for this purpose, and give a table showing concentrations of bichromate and copper sulphate corresponding to concentrations of chlorine from 0·01 to 10 parts per million. G. C. J.

Estimation of Tryptophane. E. Herzfeld. (*Biochem. Zeitsch.*, 1913, **56**, 258-266.)—The tryptophane is estimated colorimetrically or spectrophotometrically by a method based on the observation that when tryptophane solutions are treated with *p*-dimethylaminobenzaldehyde and hydrochloric acid a permanent blue colour is produced after several hours. Skatol behaves similarly, but the product formed in this case is soluble in chloroform, whilst that formed from tryptophane is not. The aldehyde solution is prepared by dissolving *p*-dimethylaminobenzaldehyde (20 grms.) in concentrated hydrochloric acid (500 c.c.) diluted with water (500 c.c.). The tryptophane solution (50 c.c.) is treated with 10 c.c. of the aldehyde solution and 100 c.c. concentrated hydrochloric acid, and allowed to stand for thirty hours. The blue colour formed is compared with that of an ammoniacal copper solution prepared by treating 1 c.c. of a 1 per cent. solution of ignited copper sulphate with 20 c.c. ammonia and diluting to 100 c.c. The colour of this solution is equal to that produced by 0·0001 grm. tryptophane. For the estimation of tryptophane in proteins the material is washed with absolute alcohol, dried, dissolved in 500 c.c. of 0·5 per cent. sodium carbonate solution, treated with 0·5 grm. pancreatin of which the tryptophane content is known, placed between layers of chloroform and xylene, and placed for twenty-four hours in an incubator. After cooling, the tryptophane is estimated as described above. If indole is present it must be removed by steam distillation. E. W.

Analysis of Turkey-Red Oil. W. Fahrion. (*Chem. Zeit.*, 1913, **37**, 1372.)—To determine the water, 3 to 5 grms. are heated in a weighed platinum dish until the cessation of frothing and formation of a small cloud of vapour show that the water has been driven off. The dish is then re-weighed. The valuation of Turkey-red oils should be based on the total fat found on boiling with hydrochloric acid and extracting with ether. An oil was thus treated, and the acid and iodine values of the separated fat determined. This fat was saponified, and total fat, acid, and iodine values again determined. Finally, the oil was saponified before treatment with hydrochloric acid, and the same three determinations carried out. The iodine numbers show that there is no hydroxystearic acid present, and that none is formed during saponification. The observed rise in fat yield and acid number is due to absorption of water, with depolymerisation of the ricinoleïc acid, but this does not take place when the saponification is preceded by a treatment with hydrochloric acid. O. E. M.

Identification of Urea and its Precipitation from very Dilute Solutions. R. Fosse. (*Comptes rend.*, 1913, **157**, 948-951.)—Urea, like certain other diamides, condenses with xanthhydrol to form dixanthylcarbamide. This compound has the formula, $CO[NH{\cdot}CH{:}(C_6H_4)_2{:}O]_2$; melting-point about 261° C., with decomposition according to the rapidity of the heating. For the identification of urea, 2 parts (by volume) of a urea solution in 70 per cent. acetic acid (and containing about 0·05 grm. of urea) are mixed with 7 parts of pure acetic acid and 1 part of a 10 per cent. xanthhydrol solution in alcohol. After some hours the precipitate is collected, washed with 10 per cent. acetic acid, then with water, dried, weighed, and the quantity of nitrogen in it is estimated. Dixanthylcarbamide contains 6·66 per cent. of nitrogen. A precipitate is obtained with as small a quantity of urea as 1 mgrm. per liter of solution; in this case the condensation takes about seventeen hours for completion. The precipitate should be separated by centrifugal action, washed with cold alcohol, then dissolved in a small volume of boiling alcohol, and allowed to crystallise. The crystals exhibit a characteristic needle-like shape when observed under the microscope. W. P. S.

Fractional Distillation of Mixed Pine Woods. J. H. Paterson and R. E. Forbester. (*J. Soc. Chem. Ind.*, 1913, **32**, 1053-1055.)—Since large quantities of wood—chiefly pine wood—have to be got rid of every year in shipyards, experiments were carried out with a view of ascertaining whether it would be possible to carbonise the wood and recover the by-products in an economical manner. The retort employed was capable of holding about 56 pounds of wood, and 23 charges of pitch pine (dried at 60° C., until it lost 10 per cent. in weight), and 40 charges of broken waste wood (dried until it lost 18 per cent. in weight) were distilled. The following are the average yields per ton of wood:

	Pitch Pine.	Mixed Pine Woods.
Turpentine oil	14 gallons	8 gallons
Tar	97 „	49 „
Acetate of lime	30 pounds	50 pounds
Alcohol and acetone	1·5 gallons	2 gallons
Charcoal	500 pounds	370 pounds

The turpentine oil had a sp. gr. of 0·846 at 17° C., and in all its properties was identical with American turpentine. The heavy tar consisted of phenols, 9·5; basic substances, 10·0; hydrocarbon oils, 36·0; and pitch residues, 25·5 per cent. The phenols consisted mainly of monophenols and cresol. The hydrocarbon oils consisted of pinene, and its homologues, together with small quantities of paraffins and higher alcohols, possessed fair drying properties, and would make good turpentine substitutes.

In a properly designed apparatus the fractional distillation of the pine woods available in England is not a difficult matter. The main objection to the process is the great length of time required to complete the distillation, owing to the bad conductivity of the material inside the retort. W. P. S.

INORGANIC ANALYSIS.

Arsenic as a Normal Constituent in Soils. G. Zuccàri. (*Gazz. Chim. Ital.*, 1913, **43**, 398-403.)—Twenty samples of Italian soil of different physical and chemical characters were taken at various depths and at altitudes from 0 to 2,400 metres above the sea-level. No artificial manures had been used on the ground whence the samples were taken. In each case arsenic was found, the quantity ranging from 0·187 mgm. (Bondo) to 6·0 mgm. (Brescia) per 100 grms. of soil. The larger quantities of arsenic were found in soils poor in iron, but no relationship could be established between the proportions of the two elements. C. A. M.

Titration of Boric Acid. E. B. R. Prideaux. (*Zeitsch. anorg. Chem.*, 1913, **83**, 362-368.)—Salm suggested (*Zeitsch. phys. Chem.*, 1911, **57**, 471) that his work with Friedenthal on a great number of indicators had put it in the power of an analyst to select a suitable indicator for the titration of any weak acid or base, and in particular he expressed the view that trinitrobenzene would serve for the titration of the third hydrogen atom of phosphoric acid. No method founded on the use of this indicator has been described, and, viewing the problem from the standpoint of the theory of hydrolysis, it is shown that the form of the neutralisation curve excludes the possibility of a sharp end-point. Similar considerations indicated that boric acid might possibly be titrated with tolerable exactness by the selection of a suitable indicator, and this has been found in tropaeoline O, of which a 0·04 per cent. solution is prepared, and about 0·5 c.c. used when the volume of the fully titrated liquid lies between 50 and 100 c.c. With pure solutions of boric acid of concentrations between $\frac{N}{4}$ and $\frac{N}{8}$, the method gives results within 1 per cent. of the truth, and it is suggested that it may serve for some technical purposes, and that, even in exact work, the first titration might be made in this way and the second with addition of glycerol in the usual manner. With pure solutions as weak as $\frac{N}{10}$, the results are about 3 per cent. too high, and with $\frac{N}{100}$ solutions 10 per cent. too high. As the solution obtained by Wherry and Chapin's modification of the distillation method contains sodium chloride as well as boric acid, the influence of this salt was studied. It has no disturbing influence, and, moreover, if the solution be saturated with sodium chloride, very dilute solutions of boric acid may be titrated with as much accuracy as stronger ones. In such cases it is advisable to use 1 c.c. of the indicator.

G. C. J.

Rapid Analysis of Bronzes. K. Schenk. (*Chem. Zeit.*, 1913, **37**, 1464.)—Tin (more or less contaminated by other metals) is separated as dioxide; copper and lead are determined electrolytically; iron is precipitated as ferric oxide; and, after removal of ammonium salts and nitrates, zinc is deposited electrolytically. The alloy (1 grm.) is decomposed by heating on the water-bath with 10 c.c. nitric acid, the excess of acid removed by evaporation nearly to dryness, and the residue treated with 200 c.c. of 1·5 per cent. nitric acid. The mixture is boiled, allowed to stand overnight, and the metastannic acid is then filtered off, washed with 1 per cent. nitric acid, ignited, weighed as SnO_2, and fused with sodium carbonate and sulphur for the recovery of traces of copper, zinc, lead, and iron. The filtrate from the metastannic acid is electrolysed hot, using platinum gauze electrodes, mechanical agitation, and a current of 2 amps. at 8 volts. After thirty minutes the electrolyte is siphoned off without interrupting the current, and the electrodes are washed with water. The cathode, with its deposit of copper, is washed with alcohol and ether, dried and weighed in the usual manner, but the lead peroxide on the anode must be dried for an hour at 180° C. The exhausted electrolyte is concentrated, iron precipitated by means of ammonia, and the precipitate filtered off, washed, ignited, and weighed. The filtrate is evaporated to dryness after addition of sodium hydroxide to decompose ammonium salts, and the residue is taken up in water and evaporated with sulphuric acid to decompose nitrates. The nitrogen-free residue is dissolved in 150 c.c. water, and the solution is made strongly alkaline by addition of 10 grms. sodium hydroxide. It is then electrolysed hot, using a current of 2 amps. at 8 volts and mechanical agitation. The use for this purpose of the cathode used for the deposition of copper saves a weighing and avoids any alloying of platinum with zinc. Since zinc is readily soluble in strong alkali, the current must not be interrupted until the deposit has been well washed. The combination of methods described is said to give satisfactory results in a minimum of time (*cf.* Gemmel, ANALYST, 1913, **38**, 388).

G. C. J.

Gravimetric Estimation of Calcium as Oxalate. S. Goy. (*Chem. Zeit.*, 1913, **37**, 1337-1338.)—When calcium oxalate is precipitated in the usual way from boiling solution, the precipitate has the composition $Ca(COO)_2.H_2O$, and this precipitate can be completely freed from adherent moisture, without loss of its water of crystallisation, by filtration on a Gooch crucible and drying for four hours at 100° to

105° C. When precipitated from a cold solution, the precipitate consists of a mixture of $Ca(COO)_2.H_2O$ and $Ca(COO)_2.3H_2O$, so that hot precipitation, dictated in ordinary circumstances by mere considerations of convenience, is for this method essential. The use of a Gooch crucible is also essential, as attempts to use ordinary tared filters showed that the precipitate lost a notable fraction of its crystal water even in three hours at 105° C., and no constancy in weight was attained in eight hours. Dried in a Gooch crucible for three hours at 100° C., precipitates may show a loss of 0·5 mgrm. after a further three hours at the same temperature, but more often show no further loss even on twenty-four hours' heating at 105° C. Attempts to find conditions under which calcium oxalate could be completely dehydrated without partial decomposition to carbonate were unsuccessful. The maximum error of the method as described is 0·4 per cent. where the quantity of oxalate weighed is 0·2 grm. and upwards. On smaller weighings the percentage error is higher, but for quantities less than 0·1 grm. the absolute error does not exceed 0·6 mgrm. G. C. J.

Titration of Calcium and Magnesium in the Same Solution. P. J. Fox. (*J. Ind. and Eng. Chem.*, 1913, **5**, 910-913.)—The method depends on the precipitation of calcium as oxalate and magnesium as magnesium ammonium arsenate, solution of the mixed precipitates in acid after filtration and washing, titration of calcium oxalate by means of permanganate in the usual manner, and then of magnesium ammonium arsenate by means of thiosulphate after addition of potassium iodide, as suggested by Meade (*J. Amer. Chem. Soc.*, 1899, **21**, 146; *cf.* also Frankforter and Cohen, *ibid.*, 1907, **29**, 1464).

The directions given below are designed to keep the error of the magnesia method small and constant. They succeed in making it approximately constant, but do not reduce it to negligible proportions. The thiosulphate must therefore be standardised on a magnesia solution of known strength, and a correction determined by titrating two portions of 25 and 50 c.c. respectively. Twice the volume used in the first titration, less that used in the second, gives the volume to be deducted from the consumption of thiosulphate in all titrations. This correction is almost independent of the actual amount of magnesia present, and is mainly dependent on the action of light on hydriodic acid under the particular experimental conditions. Its magnitude is of the order 0·5 c.c. $\frac{N}{10}$ solution. The permanganate should also be standardised on a calcium solution of known strength, and a correction for the solubility of calcium oxalate—also a constant, nearly independent of the amount of calcium present—is determined by titrating 25 and 50 c.c. portions.

The solution to be analysed, which must contain enough ammonium salts to prevent precipitation of magnesia as hydroxide on addition of a slight excess of ammonia, is treated hot with excess of oxalic acid, and dilute ammonia is then added cautiously until the reaction is alkaline to methyl orange. Within a few minutes—that is to say, before any appreciable amount of magnesium oxalate has time to separate—excess of ammonium arsenate is added to the hot solution, and this is followed by the addition, with constant stirring, of 10 c.c. of strong ammonia. When the solution has become cool, 10 to 15 c.c. more ammonia is added, and the mixture preferably left overnight, though twenty minutes' shaking insures equally satisfactory results. The precipitate is filtered, washed with ammonia water, transferred as far as possible to a flask, the portion adhering to the paper dissolved in hot dilute sulphuric acid, and 10 c.c. of stronger (1 : 1) acid added to the contents of the flask. The acid used to wash the paper should not be stronger than 1 : 10 or hotter than 100° C., or the iodine titration will be rendered difficult. The solution, if less than 75 c.c., is diluted to about this bulk, and titrated with permanganate in the usual manner. It is then cooled, and a further 25 c.c. of 1 : 1 sulphuric acid is added. About 5 grms. of potassium iodide are added, not too rapidly, lest a precipitate, which dissolves with difficulty, should separate. Thiosulphate is added rapidly until the colour is reduced to pale straw, and the flask is stoppered and set in the dark for five minutes. The titration is then completed drop by drop, without the use of starch, and the solution shielded from the light for one minute. Usually there is no return of the yellow colour, or at most one more drop is required to give an end-point persisting for one minute. If the time from introducing the first crystals of iodide to the completion of the titration be kept approximately constant (eight minutes suffices), the results are more closely concordant. The maximum error of a magnesia determination is such as corresponds to ±0·8 c.c. of the $\frac{N}{10}$ thiosulphate used. On a 50 c.c. reading this is an error of 1·6 per cent., and as the error is nearly independent of the amount of magnesia present, when using this method large readings should be aimed at. G. C. J.

New Method for Electro-Analysis of White Metals. I. Compagno. (*Atti R. Accad. Lincei*, 1913, **22**, II., 221-226; through *Chem. Zentralbl.*, 1913, II., 1703-1704.)—The novelty lies in the treatment of the residue insoluble in nitric acid. The alloy (1 grm.) is digested with 20 c.c. nitric acid (sp. gr. 1·4) for some hours at room temperature, and finally for thirty minutes on the water-bath. The solution containing the whole of the lead, arsenic, and iron, and most of the copper, is submitted to the usual methods of electro-analysis. The residue of metastannic and metantimonic acids, which occludes some copper, is treated with excess of alkali and dissolved by means of 80 c.c. of sodium sulphide solution (sp. gr. 1·225) and 0·5 grm. of potassium cyanide. Antimony and copper are deposited electrolytically, employing the apparatus described in an earlier paper (*Atti R. Accad. Lincei*, 1912, [5], **21**, I., 473), and a current of 0·15 amp., the operation being allowed to proceed overnight. The deposit is weighed, the copper redissolved in nitric acid, precipitated as cuprous oxide from alkaline tartrate solution, and finally determined electrolytically, and the antimony calculated by difference. The alkaline liquor containing the tin is diluted to 600 c.c., acidified with 120 c.c. of hydrochloric acid (sp. gr. 1·19), and boiled to expel hydrogen sulphide. Hydrogen peroxide and 20 grms. oxalic acid are then added, and the tin is deposited electrolytically. For this purpose a rotating anode consisting of a spirally wound platinum wire, 35 cm. long and 1 mm. in diameter, is employed, the cathode being constructed from a piece of copper gauze (200 meshes per sq. cm.), 6·5 × 16 cm., bent into the form of a cylinder. With 2 amps. at 3·4 to 3·6 volts, the separation of tin is complete in two hours, but quantitative results may be obtained in thirty minutes by the use of 7 amps. at 5·5 to 5·6 volts. G. C. J.

Presence of Gallium in Commercial Aluminium. C. Boulanger and J. Bardèt. (*Comptes rend.*, 1913, **157**, 718-719.)—The presence of gallium was identified spectroscopically in all the samples of bauxite and commercial aluminium examined. From one sample of aluminium it was separated by treating 1·7 kilos of the metal with hydrochloric acid, and separating the various metals (copper, arsenic, antimony, tin, lead, zinc, iron, and sodium) by precipitation with hydrogen sulphide in hydrochloric or acetic acid solution. By repeating this treatment three times gallium contaminated with a little iron was left, and by a final treatment with boiling potassium hydroxide solution pure gallium was obtained. It amounted to 0·017 per cent. of the original metal. C. A. M.

Behaviour of Lead Carbonate, Basic Lead Carbonate, and Lead Sulphate towards Alkali Carbonate Solutions. F. Auerbach and H. Pick. (*Arbeit Kaiserl. Gesundheitsamte*, 1913, **45**, 113-165.)—The authors have investigated the reactions taking place when the lead compounds mentioned in the title are treated with alkali carbonate solutions. On treating lead carbonate with potassium carbonate solution, basic lead carbonate is formed, and the latter is converted back again into lead carbonate by potassium hydrogen carbonate. This reversible reaction is shown by the equation:

$$3PbCO_3 + K_2CO_3 + 2H_2O \rightleftharpoons 2PbCO_3 \cdot Pb(OH)_2 + 2KHCO_3,$$

and an equilibrium is reached, both lead carbonates being then present. The proportion of potassium carbonate to potassium hydrogen carbonate in the solution at the equilibrium-point varies, more carbonate being present as the concentration of total alkali increases, whilst the hydrogen carbonate increases with rise in temperature. Similar results are obtained by the use of sodium carbonate solutions when the total Na concentration does not exceed 0·077 *N*. With higher concentrations and under certain conditions basic sodium-lead carbonate, $NaPb_2(CO_3)_2OH$, is formed. Sodium carbonate solutions containing varying quantities of sodium sulphate behave like sulphate-free solutions towards lead carbonate and basic lead carbonate, but the equilibrium concentration in the presence of sulphate is altered.

Lead sulphate and sodium carbonate solution do not react directly to form lead carbonate and sodium sulphate. Basic lead carbonate at first forms, then basic sodium-lead carbonate which, in the presence of a sufficient further quantity of lead sulphate, is converted into neutral lead carbonate; at the end of the reaction only traces of basic lead carbonate and a corresponding quantity of dissolved sodium hydrogen carbonate are present.

Lead sulphate and sodium hydrogen carbonate solution give lead carbonate, sodium sulphate, and free carbon dioxide according to the equation:

$$PbSO_4 + 2NaHCO_3 \rightleftharpoons PbCO_3 + Na_2SO_4 + CO_2 + H_2O.$$

The reaction is reversible and, consequently, lead carbonate suspended in sodium sulphate solution may be converted into lead sulphate by the introduction of carbon dioxide under sufficient pressure. W. P. S.

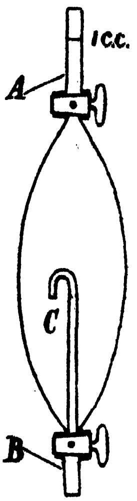
Tests of Paints for Steel, subjected to Alternate Exposure to Air and Fresh Water. P. H. Walker and S. S. Voorhees. (*J. Ind. and Eng. Chem.*, 1913, **5**, 899-905.)—The authors describe in detail the measures taken by them to eliminate all variables except the pigments. Their results confirm the pre-eminence of red lead and American vermilion as protectors of iron work. The value of the former has long been known, and American vermilion, which approximates the composition $PbCrO_4.PbO$, has been included in other published series of tests, and always taken a high place. The authors suggest that in future tests half of each test-plate should be painted with red lead paint (500 grms. red lead, 220 c.c. linseed oil, no drier) as a standard. This would eliminate any variation in conditions due to the metal. Stress is laid on the advantage of sand-blasting over pickling for preliminary cleaning of the test-plates.

Chromium oxide green paint has been gaining in favour for some time in spite of its relatively high cost, and in the tests now described proved superior to all the other paints used, except zinc yellow and the two lead paints already referred to. As used by the authors, however, it dried in an anomalous manner. The first coat dried in less than twelve hours, but the second was not even tacky at the end of several days, resembling a mixture of a pigment with a non-drying oil. It was still sticky when the third coat was applied seventeen days later. The mixture consisted of 1,760 grms. chromium oxide green, 1,800 c.c. linseed oil, and 30 c.c. of turpentine containing 4 per cent. of lead and 0·8 per cent. of manganese as linoleates.

G. C. J.

Colorimetric Estimation of Small Quantities of Manganese in Water. L. Hartwig and H. Schellbach. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1913, **26**, 439-442.)—The following modification of Marshall's method (Analyst, 1901, **26**, 195) was found to be more rapid than that described by Schowalter (*ibid.*, 1913, **38**, 477); the author agrees with Schowalter's statement that Haas's modification of the method is untrustworthy. Fifty c.c. of the water are acidified with 0·5 c.c. of iron-free nitric acid and evaporated to a volume of 25 c.c.; after cooling, an excess of 1 per cent. silver nitrate solution and 10 c.c. of 10 per cent. ammonium persulphate solution are added, the mixture is heated on a boiling-water bath until the silver chloride has settled completely (this takes about 30 minutes), then boiled for a moment, and cooled. The solution is decanted into a graduated cylinder, and the coloration is compared with that exhibited by a series of standard solutions containing varying quantities of permanganate. A standard permanganate solution is prepared by diluting 10 c.c. of a 0·287 per cent. potassium permanganate solution to 100 c.c. with a solution obtained by boiling 2 litres of water for twenty minutes after the addition of 40 grms. of ammonium persulphate and 13 c.c. of 1 per cent. silver nitrate solution. Each c.c. of this standard solution contains 0·01 mgrm. of manganese. In the case of waters containing only small quantities of chlorides, sodium chloride may be added in order to obtain a precipitate of silver chloride which will settle rapidly.

W. P. S.



Estimation of Dissolved Oxygen in Water. J. J. van Eck. (*Zeitsch. anal. Chem.*, 1913, **52**, 753-755.)—Romijn's modification of Winkler's method is in every respect more convenient than the original method, but there are cases where its use may give rise to erroneous results—for example, with sea water (*cf.* Jorissen, *Zeitsch. anal. Chem.*, 1910, **49**, 424). In these cases, the use of Rochelle salt to inhibit the formation of a precipitate must be abandoned. This involves the abandonment of Romijn's pipette with its manifest advantages. The author has now devised a pipette suitable for use according to Winkler's original method, but which, like Romijn's pipette, makes it possible to take samples from wells at definite depths or from springs difficult of access, which excludes any possibility of error due to atmospheric oxygen, and is convenient to handle. The construction is self-evident from the illustration; the capacity of the pipette, like that of Romijn, is about 200 c.c. The pipette is filled in the usual manner, the form of the bent tube *C* insuring thorough mixing. The reagents are measured in the 1 c.c. graduated tube *A*, and admitted separately by cautious manipulation of the cocks. The precipitate of manganous hydroxide soon settles to the bottom of the apparatus, and, when this is the case, 1 c.c. hydrochloric acid is quickly admitted. With a little practice this can be accomplished before the precipitate shows any sign of rising or dissolving. The precipitate is dissolved by agitating the pipette, the contents of which are then transferred to a flask and titrated with thiosulphate in the usual manner. In calculating the result, the content of the tube *C* is subtracted from that of the pipette, and a further correction of 2 c.c. is made for the volume of the reagents used. G. C. J.

Solubility of Atmospheric Oxygen in Water. T. Carlson. (*Zeitsch. angew. Chem.*, 1913, **26**, 713-714.)—The author tabulates values found experimentally by five different observers for the solubility of atmospheric oxygen in water under a pressure of 760 mm. A formula deduced from the principles of thermodynamics is also given for calculating the differences in solubility at different temperatures. In the following table the solubilities are given for pressures of 760 mm. and $760 - f$ mm., where f is the tension of aqueous vapour corresponding to the respective temperatures:

Temperature °C.	Solubility parts per 1,000 at 760 mm.	Solubility parts per 1,000 at $760 - f$ mm.
0	10·26	10·20
5	9·02	8·94
10	8·02	7·92
15	7·21	7·09
20	6·55	6·40
25	6·00	5·81

J. F. B.

Estimation of Palladium by Means of α-Nitroso-β-Naphthol and Separation of Palladium from Copper and Iron. M. Wunder and V. Thüringer. (*Zeitsch. anal. Chem.*, 1913, **52**, 737-740.)—α-Nitroso-β-naphthol, which was introduced into analytical practice by Ilinski and v. Knorre as a precipitant of cobalt and means of separating that metal from nickel, serves for the quantitative precipitation of palladium. Under certain conditions it also precipitates copper and iron, but under other conditions, easily realised in practice, it effects a sharp separation of palladium from these metals. The solution, which should not contain more than 0·05 grm. palladium, as the precipitate is very bulky, is brought to a volume of about 150 c.c., mixed with 20 c.c. hydrochloric acid and 20 c.c. acetic acid, and heated to boiling. A hot saturated solution of nitroso-β-naphthol in 50 per cent. acetic acid is added in small quantities at a time to the boiling solution. At first the solution acquires a red colour, but on continued boiling a red precipitate separates and collects above the liquid, which is no longer red. More reagent is added from time to time until further additions no longer restore the red colour to the liquid, which is now yellow, due to excess of the reagent, whether iron is present to intensify the colour or not. Filtration must be effected while the solution is still hot, and the precipitate must be washed with hot 5 per cent. hydrochloric acid, and finally with hot water. The filter and contents are dried, transferred to a crucible, heated cautiously on a hot plate in the draught cupboard until noxious vapours cease to be evolved, then with a free flame, and finally with the blowpipe. Incineration is facilitated by the addition of a few crystals of oxalic acid. The palladium sponge is reduced in a current of hydrogen and cooled in a current of carbon dioxide. In the filtrate the estimation of copper as thiocyanate presents no difficulty after neutralisation of most of the acid. Reduction with sulphurous acid never yields a colourless solution, but this is due to the nitroso-β-naphthol, and the precipitate of copper thiocyanate is sometimes not quite white, but even when distinctly yellow the error is negligibly small. In the filtrate from the copper thiocyanate, the iron is precipitated as hydroxide after oxidation by means of nitric acid.

The maximum error of the above method for palladium is 0·4 mgrm., but it possesses no advantage over the dimethylglyoxime method (ANALYST, 1913, **38**, 79). When that method is used for the separation of palladium from copper and iron, it is not necessary to separate the iron as sulphide in the filtrate from the copper thiocyanate as originally recommended (*loc. cit.*). Prolonged boiling with sufficient nitric acid will decompose dimethylglyoxime, though less readily than such treatment decomposes nitroso-β-naphthol. G. C. J.

Estimation of Periodate in Presence of Iodate and Iodide. E. Müller and G. Wegelin. (*Zeitsch. anal. Chem.*, 1913, **52**, 755-759.)—Periodate can be estimated in presence of iodate by treatment with potassium iodide in solutions in which the hydrion concentration is determined by the presence of sodium bicarbonate, with subsequent titration of the liberated iodine by means of arsenious acid. At this concentration of hydrogen ions the reaction $IO_3' + 5I' + 6H^{\cdot} \rightleftharpoons 3I_2 + 3H_2O$ (1) proceeds neither in one direction nor in the other, and only the fourth oxygen atom of the periodate reacts with iodide: $IO_4' + 2I' + 2H^{\cdot} = IO_3' + I_2 + H_2O$ (2) (Müller and Friedberger, *Ber.*, 1902, **35**, 2655).

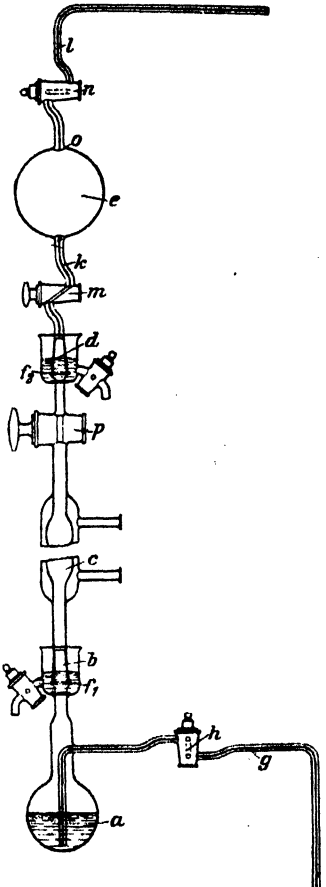
A difficulty arises in the case of solutions containing iodide and much caustic alkali. When excess of bicarbonate is added to such solutions, the result is a mixture of carbonate and bicarbonate, and the hydrion concentration is so low that reaction (2) proceeds with extreme slowness. Saturation with carbon dioxide is tedious, and the use of mineral acid or even acetic acid to neutralise the caustic alkali is out of the question, as it is impossible to conduct this neutralisation without raising the hydrion concentration locally and temporarily to a point at which iodate and iodide react.

A remedy is found in the use of boric acid. A solution of borax and boric acid inhibits reaction (1) in either sense just as bicarbonate does, and boric acid alone added to a neutral solution of iodide and iodate gives rise to no visible separation of iodine unless a very considerable time is allowed. Alkaline solutions containing periodate, iodate, and iodide are therefore treated with a saturated solution of boric acid until a drop brought in contact with phenolphthaleïn-paper no longer gives rise to a red colour. Excess of potassium iodide is added, and the iodine liberated, which is equivalent to one-fourth of the oxygen of the periodate, is titrated with arsenious acid.

The total periodate and iodate can be determined in the same portion of the solution, and by means of the same standard solution, without the use of a thiosulphate solution as is usual. After the first titration, the solution is acidified with sulphuric acid until a drop colours Congo-red paper blue. The whole of the iodate originally present, as well as that resulting from the partial reduction of periodate, now reacts with iodide. Excess of borax is added until Congo-red paper is no longer turned blue, and the liberated iodine is titrated with arsenious solution. The consumption of arsenious solution, less three times that required in the first titration, measures the iodate originally present. The test numbers are exact. G. C. J.

Radio-Elements as Indicators in Analytical Chemistry. F. Paneth and G. v. Hevesy. (*Monatsh.*, 1913, **34**, 1401-1407.)—The authors describe the method by which they determined the solubility of certain lead salts (ANALYST, 1913, **38**, 476), and point out that their results are more probably correct than earlier ones, as their method measures only the lead present, and its results are unaffected by any accidental impurities. For the solubility of lead chromate, the literature only discloses a single determination by Kohlrausch, who admitted that the method he employed only allowed the number to be roughly estimated. The authors suggest a number of analogous uses to which radio-active elements may be put—for example, the use of thorium C or radium E as indicators of bismuth. G. C. J.

Estimation of Radium in Radio-Active Substances. A. L. Fletcher. (*Phil. Mag.*, 1913, (6) **26**, 674; through *Chem. Zentralbl.*, 1913, II., 1774.)—A fragment of the mineral, weighing from 0·1 to 0·0001 grm., is heated electrically in a cavity in a thin carbon rod to a temperature of 2,000° to 3,000° C. The volume of the vessel containing the carbon rod may be varied by adjusting one of its walls. The vessel is connected on one side with a rubber balloon, which serves to take up the increase of gas-volume due to the expansion of the air and the evolution of carbon dioxide; and on the other, through calcium chloride and soda-lime tubes, to the electroscope. An estimation of the emanation can be carried out in from twenty to thirty minutes. Results on several minerals were such as might have been expected from their content of uranium. The relation between temperature and quantity of emanation set free in one minute from a small quantity of pitchblende was investigated. The curve obtained showed that below 750° C. only about 10 per cent. of emanation is given off; above this the quantity rapidly increases, being about 50 per cent. at 800° C., and 100 per cent. at 900° C. O. E. M.

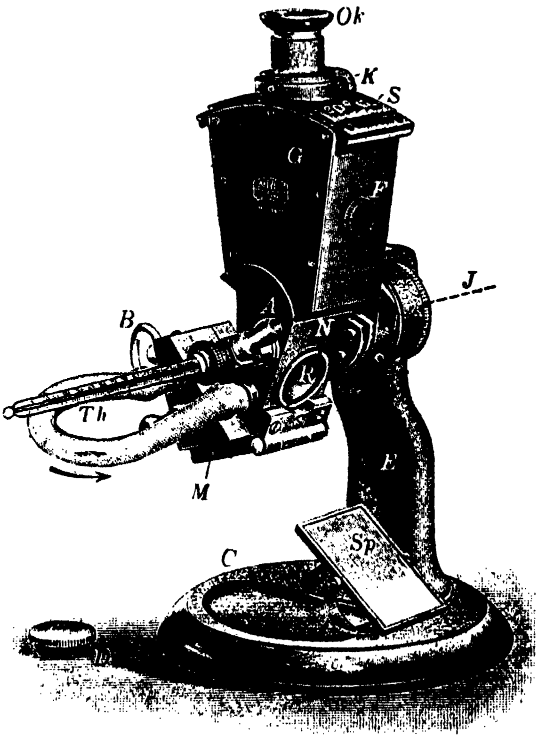


Estimation of Radium by the Emanation Method. E. Ebler. (*Zeitsch. angew. Chem.*, 1913, **26**, 658-661.)—The method previously described for the estimation of radium in minerals and rocks (Analyst, 1912, **37**, 323, 421) is not convenient for the estimation of radium in the acid and alkaline liquors which need repeatedly to be tested during the working up and concentration of ores and residues for their radium content. For this purpose, the apparatus illustrated has been devised. The (acid) liquid to be tested is placed in the hard-glass flask *a*. The reflux condenser *c* (abbreviated in the illustration) is connected to the flask by the ground joint *b*, and the contents of *a* are boiled for fifteen to thirty minutes, whilst a stream of air (filtered through cotton-wool) is drawn through *g*, *h* and the liquid by suction applied at *d*, the apparatus above *d* being disconnected at this stage. All emanation having been expelled in this way, *h* is closed, the cup, f_1, filled with mercury, the apparatus exhausted, and *p* closed. The time is then noted, and after one or more days, according to the quantity of radium present and the sensitiveness of the electrical measuring instruments available, the bulb *e* is connected to the apparatus by means of the cupped ground-joint *d*. The bulb *e* and its connections are next exhausted by connecting *l* to a pump and having *m* and *n* open, but *p* still closed. The cock *n* is then shut, *p* opened, and the contents of *a* boiled. In order to transfer the whole of the emanation to the bulb *e*, a beaker containing hot saturated brine, free from any trace of sulphate, is brought under the vertical limb of *g*, the flame is removed from *a*, and the cock *h* cautiously opened, and kept open until liquid rises up to the bore of the cock *m*. This marks the end of the "collecting time," and the period which has elapsed since the liquid was freed from emanation at the beginning of the experiment is noted. The subsequent transference of the emanation from the bulb to a suitable ionisation chamber is deferred for about five minutes to avoid interfering effects due to any thorium emanation or actinium emanation which may be present. This period suffices for their transformation to forms which do not interfere, whilst the radium emanation, with its relatively long life, is not sensibly diminished. An ionisation-chamber and electrometer are figured and described in the paper, and directions are given for conducting this part of the work, which is common to all emanation methods for the estimation of radium. In default of a solution of radium-barium chloride of known strength, a standard solution may be prepared from undecomposed uranium pitchblende, the radium content of the latter being calculated from the uranium content by multiplying the latter by $3{\cdot}33 \times 10^{-7}$. G. C. J.

APPARATUS; ETC.

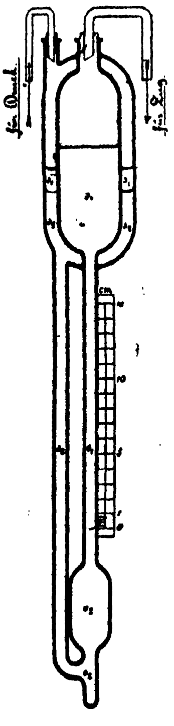
Sugar Refractometer.

F. Löwe. (*Zeitsch. angew. Chem.*, 1913, **26**, 671-672.)—During the last five years the refractometric method has come into use for the estimation of the dry solids in sugar juices and syrups. So far as the influence of the difference between the refractive indices of the sugar and the non-sugar is concerned, it has been established by A. Lange that the error due to this difference is always less than the subjective error of observation —*i.e.*, is less than 0·1 per cent. expressed as dry solids. A special refractometer, a modification of the Abbé instrument, has been designed by Professor Schönrock and the author. It comprises a double prism, *MN*, with arrangements for controlling the temperature, a compensator which enables the observations taken by ordinary light to be read off directly in terms of sodium light, and an eyepiece rotated by the milled nut *K*. The operation consists in placing a few drops of the solution in the receptacle of the prism, and turning the milled nut until the limiting line coincides with the point of intersection of two crossed threads in the eyepiece. The instrument is calibrated to read directly in terms of percentage of dry solids, the graduations being situated in the field of vision of the eyepiece. The value of the scale divisions has been determined between 0 and 60 per cent. from observations made by Schönrock, and between 60 and 85 per cent. from Main's table. The ordinary instrument is correct at 20° C., the tropical pattern is calibrated for 28° C.; for other temperatures a table of corrections is supplied. This refractometer can be utilised even with very dark-coloured, highly-concentrated syrups, being provided with an arrangement for observations by



reflected light. For this purpose the cover *D* is removed from the opening *R*, and the mirror *Sp* is adjusted so that it throws no light into the prism. Then, however dark the syrup may be, the field of vision is sharply divided into a bright and a dull half, and the line of division is made to coincide with the crossed threads of the eyepiece. Readings taken by reflected light are practically identical with those taken by transmitted light.

J. F. B.



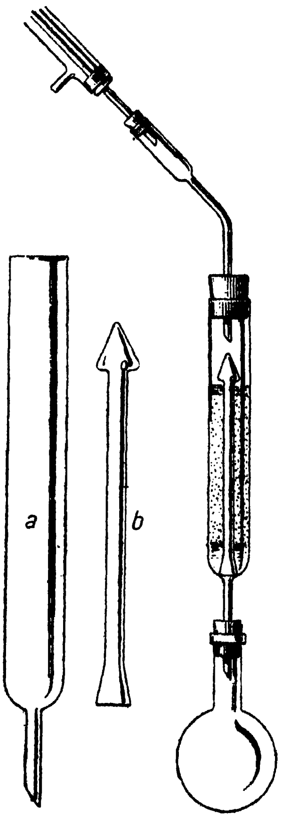
New Differential Pressure Gauge. P. Verbeek. (*Chem. Zeit.*, 1913, **37**, 1338-1339, 1361-1362.)—The author describes a differential pressure gauge embodying some novel features, whilst preserving those which distinguish König's well-known gauge from earlier and less satisfactory models. The principal improvement lies in the design of the lower portion of the instrument, which secures two important desiderata. It will be seen from the illustration that if the instrument is connected to a gas space in which the pressure is in excess of that which the scale provides for, or even if connected by the left limb to a gas space in which the pressure is below that of the atmosphere, no mixture of the liquids will take place unless the pressure or suction is quite large. König's instrument is immediately put out of order if the pressure limb is, in error, connected to a gas space below atmospheric pressure, or the suction limb to a space above atmospheric pressure. The instrument now described may be connected by either limb to a gas space at either positive or negative pressure three times as great as is covered by its scale. The concentric arrangement at the bottom of König's instrument could not be retained; but the arrangement adopted makes the zero point of the gauge so nearly independent of small variations from the vertical that, for practical purposes, it is in no way inferior to König's, which made this independence complete. Other improvements are—the substitution of rubber corks for the glass stopper of the König instrument, and the use of a centimetre scale instead of an arbitrary one. The value in mm. water of the actual readings of such an instrument tends to vary in time if the lighter liquid is a mixture, owing to evaporation of its lighter constituents, and, after recharging, the indications of the arbitrary scales of commercial instruments are sometimes 25 per cent. in error when the liquids are obtained from the makers. Though it is within the power of a chemist to prepare liquids which will make the indications exact, it is much more convenient to have a centimetre scale for which the exact factor can be determined after each charging, and if necessary at intervals irrespective of recharge, by comparison with a precise water gauge. A water gauge specially adapted for this purpose, but useful for the direct measurement of pressures beyond the range of a sensitive differential gauge of normal dimensions, is figured in the paper.

The gauge is made by Willy Manger, G. m. b. H., Dresden 16, the normal magnification being about 10, and the maximum length of scale 30 cm. By varying the dimensions of the instrument, and choosing liquids of very low and but slightly differing specific gravity, the magnification can be approximately doubled; but it is shown that this is the practical limit of an instrument of this type. G. C. J.

Determination of Melting-Points (of Fats, etc.) by the Thermometer Bulb Method. R. Meldrum. (*Chem. News*, 1913, **108**, 223-224.)—Variations in the melting-point of a fat are not due to the heating and length of the subsequent period of cooling which a fat has undergone before its melting-point is determined, but are caused by errors in manipulation. The thickness of the coating of fat on the thermometer bulb is of the first importance. The fat should be melted about 10° above its melting-point, the thermometer dipped rapidly in and out, cooled for fifteen minutes, and the melting-point then determined. If the first drop of fat that appears is turbid, the coating on the bulb is too thick; if the drop hangs too long before falling, and a second drop fails to appear, the coating is too thin. After dipping, failure to remove any drop of fat from the end of the bulb before cooling will cause the readings to be too high. In the actual determination, the temperature should not be raised at a greater rate than 1° C. per two minutes. W. P. S.

Extraction Apparatus. M. François. (*J. Pharm. Chim.*, 1913, **8**, 409-410.)—The smaller tube, *b* (see figure), is about 1 cm. in diameter; its lower end is open and slightly expanded, whilst the upper end is blown out into the shape of a small cone, a hole, 4 mm. in diameter, being made at the lower edge of the cone. This tube is fitted down the centre of the wider tube, *a*, a layer of fat-free cotton-wool is packed round the base of the smaller tube, the material to be extracted is introduced into the space between the two tubes above this layer and is covered with another thin layer of cotton-wool. The vapour of the solvent passes upwards through the centre tube, and, after being condensed, falls drop by drop through the material, passing back into the flask through the small space between the bottom of the inner tube and the outer tube.

W. P. S.



Densities of Certain Dilute Aqueous Solutions by a New and Precise Method. A. B. Lamb and R. E. Lee. (*J. Amer. Chem. Soc.*, 1913, **35**, 1666-1693.)—After discussing the weak points of the pycnometer method and of the displacement method, in which a submerged glass float is suspended and weighed in the liquid under examination, the authors describe their modification of Pisati and

Reggiani's submerged sinker method (*Rend. Reale Accad. dei Lincei*, 1890, [4], **7**, 99). Its one drawback lies in the difficulty of varying the buoyancy of the sinker by small amounts, and if this is to be effected by means of added weights, these would have to be as small as a hundredth of a milligramme if it were desired to obtain an accuracy equal to that obtained by Kohlrausch (*Wied. Ann.*, 1894, **53**, 14) by his displacement method (viz., 1 part in 5 millions). Some immaterial, easily variable, and measurable external force is wanted which will produce displacements of the sinker in a vertical direction, and thus fulfil the function of added weights; and this force was found in the electro-magnetic attraction exerted by means of an electric current sent through a properly placed external circuit upon a piece of soft iron enclosed in the bulb of the sinker. The electro-magnetic pull was only employed to produce the finer shades of variation in buoyancy, platinum milligramme weights being added to or taken from the float where larger variations were necessary. With the sinker weighted so as just to float in the liquid, a current is passed through an insulated copper wire helix, attached to the bottom of the outside of the beaker, just sufficiently strong to cause the float to descend in the beaker, and to hold the platinum point with which it is provided in contact with a little platinum saucer at the bottom of the water. When this position had been maintained for a time long enough to allow all eddies in the liquid to disappear, the current in the helix is slowly diminished until the platinum point of the float just leaves the platinum saucer. The current reading at this point is then carefully noted on a milliammeter in the circuit. This procedure was first carried through with pure water, and then a known quantity of stock solutions from the salt under investigation was added to the water, and the same process repeated. From the weight added to the sinker, the weight equivalent of the current used and the known weight and volume of the sinker, the density of the solution with reference to water can be calculated. A full description of the apparatus employed is given, together with a discussion of all possible errors, and the corrections to be applied for them. The original paper must be consulted for these, and for the very numerous results obtained when working with solutions of seven different salts in dilutions varying from $\frac{N}{0\cdot0001}$ to $\frac{N}{0\cdot01}$. A probable error of less than 1 unit in the seventh decimal place is claimed for the results. The variations in the volume changes during solution indicate in general a progressively increasing compression either of the water or of the solute or both, as the dilution becomes greater; but an approximate constancy is reached at dilutions when the electrolytic dissociation is practically complete. Of the solutions studied (magnesium sulphate, sodium chloride, potassium chloride, lithium chloride, ammonium chloride, zinc sulphate, and sodium carbonate), the last mentioned alone presented an anomalous behaviour in this particular, for in very dilute solution the contraction became less rather than greater; but it has been shown that this can be explained on the basis of the unusual contraction which is found to occur during the neutralisation of carbonic acid, proving that the progressive hydrolysis of sodium carbonate, as dilution increases, may indeed be the reason for the anomaly.

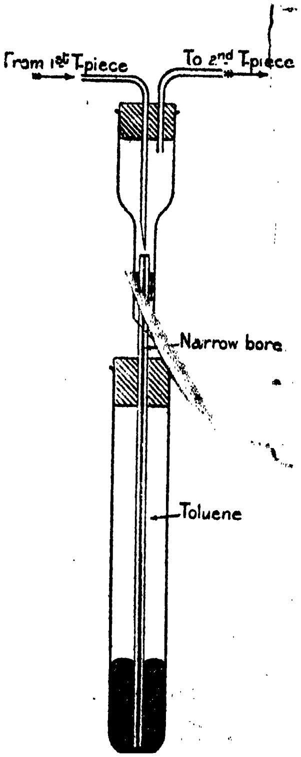
H. F. E. H.

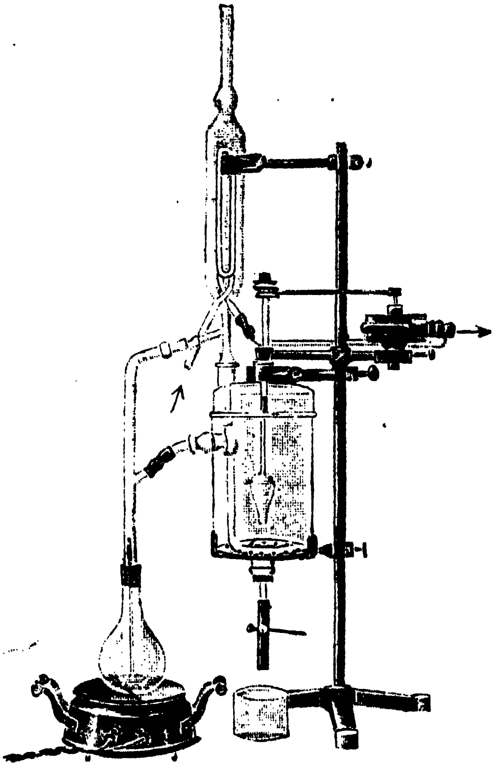
Improved Type of Calorimeter to be used with any Calorimetric Bomb.

J. A. Riche. (*J. Amer. Chem. Soc.*, 1913, **35**, 1747-1750.)—The paper deals with an attempt to eliminate the errors usually met with in such work by the use of a vacuum cup with a suitable covering. The bomb, which may be of any type, is contained in the vacuum cup containing water. The vacuum cup is 14 cm. wide, 30 cm. high, inside measure, and is obtainable from the Vereingte Fabriken f. Laboratoriumsbedarf, Berlin. The stirrer is of the screw propeller type, which is more efficient than any other. The bomb used by the author is of the Kroecker style. The vacuum cup is surrounded by an oak box lined with 1 inch of pressed cork glued to the wood. The bomb containing the material to be burned is charged with 30 to 40 atmospheres of oxygen and is then placed in a split-ring and the wires connected. The water in the vacuum cup is brought to any desired temperature within plus or minus 5° C. of the room temperature. Temperature equilibrium having been established after the ignition, the bomb is removed, opened, and rinsed out with water, the rinsings being titrated with $\frac{N}{10}$ alkali for nitric acid, using litmus as indicator. Ignition of the substance in the bomb is effected electrically, the platinum wire within the bomb itself being connected with the substance to be burned by means of a linen thread of sufficient size to secure ignition. A current from three 2-volt storage cells is employed, first passing through a 3-ampère fuse wire to the platinum wire just mentioned, thus assuring the constancy of the heat of ignition. H. F. E. H.

Simple Thermo-Regulator. J. G. Boyd and H. M. Atkinson.

(*Chem. News*, 1913, **108**, 248.)—A stout test-tube about 6 inches by $\frac{3}{4}$ inch is fitted with a good wooden cork through which passes a narrow-bore glass tube, fitting by means of a rubber band into the narrow part of a tube as shown in the figure. A double-bored stopper fitted with right-angle tubes closes the top of the upper portion; one of the right-angle tubes ground off like the stem of a funnel, only more acutely, on a piece of carborundum, reaches down inside almost to the top of the narrow-bore tube. Mercury is poured into the lower tube, then water, or, better, toluene, sufficient completely to fill it, a little mercury being allowed to cover the top of the capillary. By raising or lowering the gas-entry tube, any desired volume of gas can pass through. To prevent the Bunsen flame striking back when low, two glass T-pieces are employed to provide an independent gas-supply; these are controlled by a screw clip. H. F. E. H.





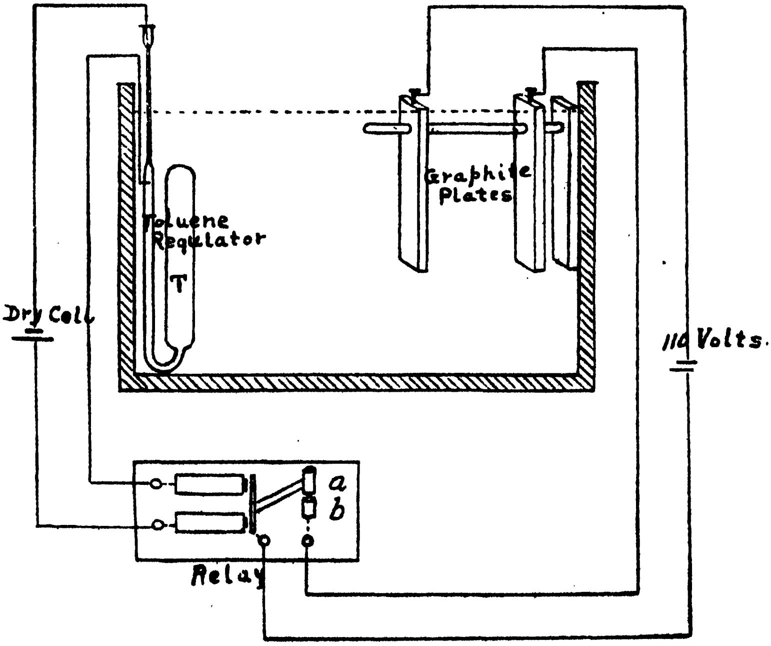
Modification of Zelmanowitz's Apparatus for Extracting Liquids.

(*Chem. Zeit.*, 1913, **37**, 1381-1382.)—In the apparatus shown in the figure, the liquid to be extracted is placed in the large vessel, and is kept in motion by the mechanical agitator. The ether is heated in the small flask, preferably by an electric heater, and the condensed vapours pass down the tube to the bottom of the extraction vessel, and escape in drops through the perforations in the ring. When the ether on the surface of the liquid reaches a certain level, it is siphoned through the side-tube back into the small flask. The apparatus may be obtained from Tolmacz and Co., Luisenstrasse 59, Berlin.

C. A. M.

Method for Electrolytic Heating and Regulation of Thermostats. I. H. Derby and J. W. Marden.

(*J. Amer. Chem. Soc.*, 1913, **35**, 1767-1769.)—The relay *ab* is a modified telegraph pony relay of 20 ohms resistance, which serves to make and break the heating circuit as the temperature of the bath fluctuates. As metal contacts are unsatisfactory for higher currents, these were replaced by carbon pencil contacts 1 to 2 cm. in diameter, one being attached to the armature, and the other to the



base. The relay coils were connected in series with a mercury-toluene regulator, *T*, in which nickel wire is used to make contact with the mercury. A single dry cell serves to operate the relay. Copper electrodes were at first used, but proved unsatisfactory owing to continuous corrosion. On substituting graphite plates no further trouble was experienced, whether a direct or alternating current was employed. The carbon electrodes were 1 cm. thick, and of two sizes, 10 × 10 cm. and 10 × 20 cm., and were fastened to wooden supports in such a way that the distance between them could be easily varied. In no case was the liquid directly between the plates observed to vary more than in other portions of the bath. For the electrolyte, tap-water and solutions of sodium sulphate were used. Vigorous stirring was found essential to good regulation. H. F. E. H.

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REVIEWS.

La Catalyse en Chimie Organique. By Paul Sabatier. Pp. 255. Librairie Polytechnique, C. Béranger, Editeur; Paris and Liège, 1913. Price 12·50 f.

This work gives a systematic survey of our present knowledge of catalysis as applied in organic chemistry, and, though not having any very direct connection with analytical chemistry, it is conceivable that several of the reactions therein described may, in the future, be put into practice by analysts.

First comes a brief account of those substances which are capable of playing the part of catalysts, followed by a more detailed description of the important types of reactions brought about by their aid, among which may be mentioned oxidation, hydrogenation (addition of hydrogen), polymerisation, condensation, dehydration, and decomposition.

In discussing the mechanism of catalysis, the author expresses the opinion that the theory which assumes the formation of unstable intermediate compounds is the only one whereby it is possible to offer a general explanation of such reactions. While it is true that this sometimes necessitates the consideration of hypothetical substances, to which many chemists offer decided objection, this theory has been his beacon-light throughout all his researches, and, despite its imperfections, must be considered good, because of its fruitfulness and because it makes it possible to predict the course of a variety of reactions.

The book, written in a clear and concise manner, is obviously the work of a man whom the chemical world rightly recognises as a "master of his subject."

Arthur W. Crossley.

Merck's Reagenzien-Verzeichnis. Dritte Auflage. 1913. Pp. 446.

This third edition of Merck's well-known list of reagents used in analysis has been brought up to date, and now contains 446 pages, an increase of 120 pages over the last (1908) edition. The arrangement in the volume under review is the same as in the last edition. In the first 394 pages we have a full description of practically all the most important reagents in general use in chemistry, biochemistry, microscopy, etc. These are given under the author's name, and, in order to enable the reagents for the identification of any substance to be readily found, there is also a complete alphabetical list of substances, the tests for which are given in the first part, together with the names of the tests by which the substances are identified.

As a compilation of all the most important reagents used in analysis, and more especially biochemical analysis, this work is very complete, and will prove of very great value to those engaged in work of this nature. H. R. LE SUEUR.

DISINFECTION AND DISINFECTANTS. By M. CHRISTIAN. London: Scott, Greenwood and Son. 1913. Price 5s. net.

This book is a translation from the German by C. Slater. It is divided into four chapters, which deal respectively with introductory matter, physical, chemical, and combined systems of disinfection. It is disappointing to find that the standardisation of germicidal power of disinfectants, on which there has recently been a considerable amount of work done, is not dealt with, the whole subject of disinfectant-testing being somewhat curtly dismissed by a reference to Koch's early work in a paragraph. The series of tar acids which form the basis of the emulsified fluids, now so extensively used, are also very briefly described. However, this little book affords a rapid survey of the properties and action of most disinfectants, the section devoted to "physical disinfection" being especially good. S. RIDEAL.

WATER PURIFICATION AND SEWAGE DISPOSAL. By DR. J. TILLMANS, Chemical Director of the Institute of Hygiene, Frankfort-on-Main. Translated by HUGH TAYLOR, M.Sc., University of Liverpool. Pp. 143. With twenty-one illustrations. 8vo. Constable and Co. 1913. Price 7s. 6d. net.

This is avowedly a compilation, mainly from German sources, though fair credit is given to other countries, and almost adulation to our own. For instance (p. 5): "For sand filtration we have to thank the Englishman *James Simpson*, who in 1829 constructed the first filter of this type;" also (p. 85): "England—as the premier country in the world—is the classic country for sewage disposal." Yet, although names and addresses of German private firms are too frequently advertised in the text, the names of such pioneers as Cameron, Dibdin, and Scott Moncrieff, are ignored in the description of their processes. Nearly all the British information relies on "the English authority Schiele" (a manufacturer, p. 106), whose name is continually repeated in the sewage section.

The limits of the book—62 pages for water, and 71 for sewage—are not sufficient to do justice to either subject. Much of the information, particularly on industrial disposal, is useful, but too scanty. Room for much more detail could have been obtained by judicious compression. Sewage disinfection is dismissed in the space of one page at the end. Some of the expressions need revising: (Ozone) "is a good *medium* for sterilising waters" (p. 20); "no filters are completely germ-tight" (p. 63); "the Main shows an average *alkalinity* of about 3 c.c. of normal *acid* per litre" (p. 66); "the more *resistible* spores" (p. 23). The illustrations and printing are well done, but there are some errors, such as *Trindall* (p. 20) for Tindel, and *Sour-Krout* is not the orthodox spelling. S. RIDEAL.

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THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

SULPHURETTED HYDROGEN FROM ARTIFICIAL GRAPHITE.

By W. H. WOODCOCK and BERTRAM BLOUNT, F.I.C.

(*Read at the Meeting, December* 3, 1913.)

A SAMPLE of massive graphite manufactured by Dr. Acheson was found to possess the unusual property of evolving sulphuretted hydrogen when broken or rubbed. The mere breaking of the graphite, so as to obtain a fresh surface, was sufficient to obtain a decided odour, whilst the scraping of the material with a knife made it smell strongly of H_2S. The sample itself was very soft and "greasy" to the touch, and it contained 0·11 per cent. total S. and 0·20 per cent. of ash, consisting of:

	Per cent.
Si	0·052
Al	0·014
Fe	0·012
Ca	0·029
Mg	0·008

An examination of the graphite was made to find the cause of this curious property, and the most reasonable suggestion seemed to be that the material occluded sulphuretted hydrogen. The investigation was therefore started from this point of view.

A small piece of the graphite was allowed to stand in the air until it became free from smell, and then heated in a combustion tube in an atmosphere of pure hydrogen, any sulphuretted hydrogen evolved being swept over and absorbed in a solution of lead acetate. After heating for a couple of hours at 300° to 500° C. without finding any trace of sulphuretted hydrogen, the temperature was raised to a red heat. No sulphuretted hydrogen could be found after one hour at this temperature. The experiment was repeated with lead paper instead of the solution, as being more sensitive, but no sulphuretted hydrogen could be detected.

Some of the original sample was very finely powdered and allowed to stand in the open until it was free from smell. This was heated in hydrogen in a similar manner to the lump. No sulphuretted hydrogen was evolved. This fine powder, after heating, did not evolve any sulphuretted hydrogen on further grinding in an agate mortar, whereas the lump of graphite used in the previous experiment, on scraping deeply, gave a fairly strong smell of sulphuretted hydrogen.

A miniature ball mill was made from a glass bottle and a number of glass marbles. A quantity of graphite, in small lumps, was placed in the bottle, and in order to prevent oxidation of the sulphuretted hydrogen, the air was swept out with hydrogen. A fair quantity of sulphuretted hydrogen was liberated on grinding the graphite. (This little mill was excellent until the bottle and the balls became coated with graphite, when, owing to slipping, the mill was useless until baffles were put in.)

Microscopical examination of the material was made, using both broken and cut surfaces. The appearance was much the same as ordinary soft graphite.

Sections etched by means of nitric acid, fused potassium chlorate, and by contact with a plane surface of boiler plate at 1100° to 1200° C. yielded surfaces which showed no definite cavities in which the gas might have been lodged.

The result of these experiments showed that the sulphuretted hydrogen could not have been occluded, and that the explanation had to be sought in some other quarter.

The mode of manufacture of the graphite is such that any sulphur present must be as sulphide, and the total sulphur in the sample (0·11 per cent.) is ample to account for everything. From its stability on heating, and its instability on exposure to ordinary air, it is reasonable to suppose that the sulphur is present as a sulphide of the class of Al_2S_3, and on these lines the following experiments were made:

Some of the graphite was placed in a stout tube fitted with a perforated cork, through which a glass rod could be pushed up and down. A strip of filter-paper moistened with a solution of lead acetate was placed just below the cork. When the graphite was pounded with the rod, sulphuretted hydrogen was liberated, as shown by the blackening of the lead acetate. When, however, the experiment was carried out in a dry atmosphere, no evolution of sulphuretted hydrogen could be detected.

On boiling some of the graphite with dilute hydrochloric acid, a trace of sulphuretted hydrogen was liberated, but on breaking it up, and so providing access to the enclosed sulphide, considerably more sulphuretted hydrogen was evolved.

A quantitative experiment was made to determine the quantity of the sulphide decomposable by water, and also of the sulphide which requires acid for its decomposition. Fifty grms. of the graphite in one piece was broken up whilst completely immersed in water, the sulphuretted hydrogen being absorbed as soon as evolved from the unstable sulphide. The water was changed from time to time as the pounding proceeded, and the sulphuretted hydrogen in solution determined by distilling in a neutral atmosphere into a solution of lead acetate.

The broken-up graphite was then digested with dilute hydrochloric acid, and the sulphuretted hydrogen evolved from the more stable sulphides distilled over into lead-acetate solution and determined. From the sulphides decomposed by water, the $S = 0{\cdot}006$ per cent., equivalent to 0·012 per cent. Al_2S_3. From sulphides not decomposed by water the $S = 0{\cdot}058$ per cent.

From these results we are led to the conclusion that the cause of the liberation of the sulphuretted hydrogen is the presence of an unstable sulphide of the type of Al_2S_3 which decomposes in the presence of moist air, and which is protected by the graphite until it becomes exposed by breaking or scraping the material.

DISCUSSION.

Mr. W. J. A. BUTTERFIELD said that he had hoped to hear the authors draw attention to the extreme sensitiveness of the nose in detecting such minute quantities of sulphuretted hydrogen as were here evolved. From comparative tests he had found the nose to be more sensitive than lead acetate paper as ordinarily used. The sensitiveness of lead-acetate paper really depended upon the local concentration of the sulphide on the paper, and if the area of the latter was reduced to a minimum relatively to the total volume of sulphuretted hydrogen in the gas brought in contact with it, as little as one volume of sulphuretted hydrogen in ten million volumes of gas could be detected, and that proportion could also be readily detected by the nose.

Dr. R. LESSING asked whether the sample upon which the authors worked was supposed to be representative of Acheson graphite. Although he should have expected this graphite to be free from sulphur, he was surprised to hear that absolute purity from mineral matter was claimed for it, because in the process of manufacture small quantities of oxides were added to the carbonaceous substance. He should like to hear how the necessary close contact between the mild steel and the graphite was brought about in the etching process.

Mr. H. F. KNIGHT remarked that, unless some special precautions were taken, the etching might be an air etching, and not an etching due to absorption of carbon by the steel.

Mr. WOODCOCK, in reply, stated that the authors were in full agreement with Mr. Butterfield's remarks. A solution of lead acetate was first tried, but afterwards the more delicate test of a spot of the solution on a piece of filter-paper was used. With regard to the question of purity, Dr. Acheson relies upon the volatilisation of all the ash constituents. In the etching process the surface of the graphite was cut down as smoothly as possible, and then pressed into contact with a piece of dense, smooth boiler plate. The effect, however, was *nil*, there being no cavities visible where the gas could have been occluded.

* * * *

THE RATE OF LIBERATION OF HYDROCYANIC ACID FROM LINSEED.

BY S. H. COLLINS, M.Sc., F.I.C., AND H. BLAIR.

(*Read at the Meeting*, *December* 3, 1913.)

IT is fairly generally known that some samples of linseed and linseed cake readily give off hydrocyanic acid on being moistened, in spite of the fact that these foods are generally regarded by farmers as safe for stock.

Some years ago a case of poisoning occurred in Westmorland which was attributed to a particular sample of linseed cake, this yielding more hydrocyanic acid by steam distillation than was usual. In some preliminary experiments we found that some samples of pure linseed yielded nearly as much hydrocyanic acid as the linseed cake which had been suspected. Hence a reconsideration of the subject became necessary.

If hydrocyanic acid were liberated in the alimentary canal of any animal, the rate of production would have a most important bearing on the subject, since, if produced slowly enough, the animal would remove it. We therefore attempted to measure the rate at which hydrocyanic acid is liberated from linseed under various conditions.

Our chief object was to imitate digestive conditions; that is, to digest the linseed with water, with and without acids, etc., and to remove the end products as they would be removed in actual digestion. For this purpose we removed the hydrocyanic acid and other volatile products by a stream of inert gas. Considerable experimental difficulties arose; finally a current of hydrogen, at the rate of 10 litres per hour at a temperature of 45° C., was employed. Air was also tried, at other velocities and other temperatures, but the best method is that of passing 10 litres of hydrogen at 45° C. The hydrocyanic acid carried over by the hydrogen was absorbed in weak sodium hydroxide. From time to time the sodium hydroxide was removed and tested for cyanides by the prussian-blue test. The colours obtained were compared with standards.

When linseed and water are treated by this method, one particular sample of Calcutta linseed which we examined yielded 0·380 mgrms. of HCN per grm. of linseed, with a probable error of ± 0·004. The velocity of reaction was such that one-half of the total amount was yielded in forty-six minutes, whereas solutions of hydrocyanic acid in water yielded one-half the total amount in twenty-six minutes, reaching in three hours' time from 98 to 99 per cent. of the total amount present. When the linseed was acidified to represent digestive conditions, no hydrocyanic acid was liberated, and similar negative results followed the use of pepsin and rennet in acid solutions. Even hydrochloric acid of $\frac{N}{100}$ strength prevents the enzyme from working, and $\frac{N}{1000}$ strength produces a marked slowing in the rate of liberation of hydrocyanic acid from linseed.

Physiological authorities state that the acidity of the human stomach is about $\frac{N}{20}$, the acidity being greater in dogs. We found that the acidity of fresh green grass is of about the same order. Under ordinary circumstances, therefore, linseed cannot produce hydrocyanic acid when fed to non-ruminant animals, because the acid in the stomach prevents the enzyme in the linseed from working.

The case of ruminants, however, presents more difficulties. For most of the stock on a farm, 2 or 3 pounds of linseed cake, with at least ten times as much dry matter in the other foods, would represent a fair daily allowance for cattle. Such a feed would provide from 5 to 10 grains of hydrocyanic acid if a linseed cake fairly rich in cyanogenetic glucosides were used. Of course, such a dose, if generated quickly, would be a fatal one; but the poison is only developed slowly. Under the ideal conditions of the experimental methods described above, one-half, or 2·5 to 5 grains, would be developed in forty-six minutes. If the linseed were divided into two meals, these quantities would be reduced to 1 or 2 grains. If, further, a slight acidity were developed in the rumen by fermentation, or the foods consumed with the linseed were in any way acid, or contained fibre or sugar, then the rate of formation of hydrocyanic acid would be reduced. The chances of poisoning, however, depend not only upon the rate of formation, but also upon the rate of removal of the hydrocyanic acid.

In the act of rumination the food is brought up into the mouth, and any small quantities of hydrocyanic acid that have developed would have a good chance of evaporating into the air. Further, every time a " bolus " is swallowed, air is forced into the rumen, and every time a " bolus" is regurgitated the same quantity of air is removed from the rumen. The food in the rumen is, in short, evaporated by air in somewhat the same way as the linseed in our experiments was evaporated by a current of hydrogen gas. The chance of poisoning cattle when fed with dry linseed, therefore, seems indefinitely remote, nor do we know of any actual case in which a full-grown cow or bullock could be said with certainty to have suffered from prussic acid poisoning as a result of the consumption of dry linseed or linseed cake.

The use of linseed for calf food presents a further difficulty. For calf rearing, a linseed " mash " or soup is often used. Such a mash is generally made by pouring boiling water on to the ground linseed, stirring up, and allowing to cool. Sometimes the mash is boiled on the fire, and sometimes linseed-cake meal is used instead of ground linseed.

By inquiry into actual cases of calf-poisoning, we came to the conclusion that the mode of preparing the mash was an important factor.

When linseed is treated with a large bulk of water at 100° C., and kept at that temperature for about half an hour, the enzyme is destroyed and no hydrocyanic acid is produced when evaporated in an atmosphere of hydrogen at a temperature of 45° C.

When linseed is treated at temperatures between 60° and 90° C. in the presence of much water, the evolution of hydrocyanic acid is checked, at 60° C. only slightly, at 90° C. almost completely. When, however, linseed is heated dry to 100° C. for half an hour previous to digestion, not only is the evolution of hydrocyanic acid not checked, but is actually increased. Very fine grinding in a mortar previous to digestion also increases the evolution of hydrocyanic acid.

If linseed cake meal is made into a pill or dumpling with water, then heated to

100° C., the hydrocyanic acid comes off slowly at first; but as soon as the pill is broken up, the hydrocyanic acid comes off rapidly, showing that in the presence of little water, heat has no destructive effect on the enzyme.

When linseed mash is made for calves, lumps will often be formed, which will behave as in these experiments. Should several calves be fed from the same pail, the last calves fed will get the largest share of these lumps. In those cases which we have investigated the reason for the calves having died seems to be that the water used was not quite boiling and that lumps were formed. Under these circumstances the heat did not injure the enzyme in the lumps, but on the contrary facilitated the formation of hydrocyanic acid. This hydrocyanic acid was mechanically confined in the lumps, and was liberated quickly in the calves' stomachs as soon as the movements of the stomach broke up the lumps.

As regards linseed cake, we find that extractives, such as petrol, ether, chloroform, etc., do not produce a very marked effect on the enzyme. The removal of the oil concentrates both enzyme and glucoside, and therefore linseed cake is more liable to be poisonous than the linseed from which it is made.

DISCUSSION.

Dr. G. D. LANDER said that he was one of the first, he believed, to be alarmed at the possibility of death being caused by linseed. He had seen sheep which, on post-mortem examination, showed distinct evidence of the presence of hydrocyanic acid in the visceral contents, and in 1910 he made a large number of feeding experiments with various kinds of linseed cake, which yielded, on the average, about 0·025 per cent. of available hydrocyanic acid. Although, however, there was on the face of it strong reason to suppose that poisoning would occur, these feeding experiments yielded no such result. Experiments were then made with a heifer about seven months old, to ascertain what was the poisonous dose of hydrocyanic acid when administered to cattle in the form of potassium cyanide. Increasing quantities were given for a considerable period, and the animal only succumbed when the dose reached 30 grains of hydrocyanic acid—*i.e.*, 60 grains of potassium cyanide—the previous day's dose having been 22½ grains. Dr. Auld, in a paper on the subject published in a Wye College Report, had suggested that the ability to withstand such doses might be due to tolerance or habituation, but in the case of hydrocyanic acid this was not to be expected on ordinary physiological grounds, seeing that the substance was so readily eliminated and therefore rapidly decomposed. The results of these experiments led him to be emphatically of opinion that to raise any serious question as to the probability of cyanide poisoning by linseed cake was merely "flogging a dead horse." At the same time, with some cakes which he had been told by Dr. Voelcker were East Indian cakes, and which yielded as much as 0·055 per cent. of hydrocyanic acid, there might be some chance of poisoning occurring. These remarks bore upon the practical aspect of the question, but thanks were due to the authors for their study of the actual rate at which the hydrocyanic acid was evolved. Their views as to the reduced chances of danger by reason of elimination appeared to coincide with his own experience. In the experiment in which a fatal effect had been produced by 30 grains of hydrocyanic acid, the dose was given in the form of a ball, and its power of exerting a toxic action would be much greater than if it had been spread over a comparatively large amount of more or less bulky food.

Mr. W. BACON suggested that in the case of a healthy animal the hydrocyanic acid was probably absorbed into the blood, and there decomposed by oxidation into ammonia and formic acid, practically as quickly as it was given off from the food; whereas in the case of an animal not in good health the process of oxidation might not go on so rapidly, and might leave some of the hydrocyanic acid free to produce a poisonous effect.

Mr. J. GOLDING said that in a case in Nottinghamshire, in which injury to calves had followed the consumption of linseed cake which yielded a rather high percentage of hydrocyanic acid, it was found that the cake had been put into a dirty pail with water and left standing overnight, the injurious effects having probably been due to contamination in the pail. On investigating the process of manufacturing the cake at the mills, he had come to the conclusion that the temperature to which the linseed was heated in the "kettle" before being crushed would ordinarily be sufficient to destroy the enzyme, though it was possible that sometimes, on Monday mornings, for instance, before the kettle was thoroughly hot, the seed might not be so strongly heated. He should like to hear whether the authors had made any determination of the hydrogen ion concentration at which this enzyme acted. Apparently the optimum concentration lay within somewhat narrow limits.

Mr. E. R. BOLTON remarked that it would be instructive to ascertain what variations occurred, in regard to the quantity of hydrocyanic acid yielded, in cakes which had been heated to different temperatures before crushing, and also the quantity of moisture in the kettle at the time of heating. Some makers added a certain quantity of water, in order to help the oil to come out more easily at the second pressing. It had been suggested that cakes made in the Anderson press were particularly likely to give off hydrocyanic acid if the seed was not preliminarily heated, but, as a matter of fact, in the Anderson press the friction caused quite enough heat.

Dr. E. K. RIDEAL remarked that hydrocyanic acid in the form of potassium cyanide had a toxicity less than that of hydrocyanic acid itself. The cyanide was not the potassium salt of hydrocyanic acid—*i.e.*, KCN—but possibly KNC. In the case of the silver salt, at any rate, there was fairly strong evidence that the molecular structure was different, and the toxic effect was also different. Possibly the decreased sensitiveness of older animals was due to the fact that their saliva was more alkaline than that of young ones.

Dr. LANDER agreed that potassium cyanide differed considerably in toxicity from hydrocyanic acid itself, a fact which had been verified by experiment. The manner in which the hydrocyanic acid was administered also made a considerable difference. For instance, if injected into the lung cavity it would be more toxic than if taken through the stomach. It might be added that in toxicological practice it was desirable to examine the fresh brain substance, hydrocyanic acid in which was always a better diagnostic guide than its presence in the alimentary tract.

Mr. COLLINS, in reply, said that some of the points mentioned in the discussion were already under investigation, but it would be a little premature to give the results now. He did not think that the matter would be much affected by the degree of ripeness of the seed, unless it were so unripe that nobody would buy it. The question of the effect of the temperature of cooking was being investigated. They had not gone into the question of hydrogen ion concentration, having been obliged to confine their attention to the more practical aspects of the matter. He had met with cakes yielding as much as 0·05 or 0·06 per cent. of hydrocyanic acid, and undoubtedly large variations occurred.

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THE ESTIMATION OF SULPHUR IN RUBBER.

By HENRY P. STEVENS, M.A., Ph.D., F.I.C.

(*Read at the Meeting, December* 3, 1913.)

The method commonly employed for the estimation of sulphur in rubber products consists in the oxidation of the sulphur to sulphuric acid, and the subsequent estimation as barium sulphate. The usual process (Henrique's) consists of the treatment of the rubber in an open vessel with nitric acid, the evaporation of the product in the presence of basic matter, followed by a fusion. The melt is then taken up with water and the sulphuric acid estimated. The process is a wearisome one, and losses occur in spite of all precautions. More recently Spence (*J. Ind. and Eng. Chem.*, 1912, **4**, 6) has recommended electrolytic oxidation in nitric acid without subsequent fusion, and has published figures which show a close accordance between the electrolytic method and Carius's method. Owing to the large volumes of gas given off during the oxidation with nitric acid, it is advisable to open the Carius tube before placing in the furnace, or to allow the first reaction between the rubber and nitric acid to take place before sealing the tube. Spence quotes (*loc. cit.*) an analysis of hard rubber, to show that this latter modification of Carius's method does not affect the results.

It is apparent that the main problem is the rapid and complete oxidation of the sulphur to sulphuric acid without loss. In the course of numerous analyses of soft rubber goods, the author found that it is possible to complete the oxidation with nitric acid without resource to electrolysis if the heating be sufficiently prolonged. Simple oxidation with nitric acid is the process commonly employed for the estimation of the sulphur in the acetone extract from rubber. It would, however, appear that a loss of sulphur may take place in all such oxidation processes in which nitric acid is employed. This has been noted by various observers, and Bertrand (*Jahrbuch der Kautschuk Industrie*, 1909, 353) arrived at the conclusion that sulphur can be lost in the form of sulphur dioxide. Potts ("Oxidation of Sulphur by Nitric Acid," "The Rubber Industry," p. 336) confirms the loss of sulphur, and appears to favour Bertrand's view that such loss probably occurs in the form of sulphur dioxide. By oxidising small quantities of pure sulphur in a beaker covered with a clock-glass, Potts observed losses up to 12·5 per cent., but by very cautious oxidation at as low a temperature as possible the loss was reduced to 2 or 3 per cent. By carrying on the oxidation in a sealed tube no loss occurred, and figures of 101·1 and 100·1 per cent. were obtained.

The author has frequently noted small losses of sulphur during oxidation, not only in the case of acetone extracts, but also of total sulphur in both vulcanised and unvulcanised mixings. This loss takes place even when the oxidation is carried out very gradually. The ordinary process was first modified by carrying out the oxidation in the presence of chlorine by adding small quantities of potassium chlorate at the beginning of the reaction and from time to time during the oxidation process, so as to keep the flask full of yellow gas while the oxidation proceeded. The results showed considerable variation, and in many cases little or no improvement. Thus a compound vulcanised in thin sheet from between plates, and containing 7 per cent. of sulphur, yielded the figures 6·88, 6·59, 6·97, and 6·74 for percentage of sulphur. The author therefore carried out the oxidation of rubber in a closed vessel, absorbing the issuing gases in a solution of caustic potash. The apparatus was of a simple character, and consisted of a 500-c.c. flask provided with a glass stopper and side-tube. The latter was fused into the side of a glass-stoppered U-tube, which connected again with an inverted bell-shaped orifice which could be arranged to dip into a beaker. In the U-tube and beaker were placed a strong solution of caustic potash, sufficient to neutralise and absorb not only any sulphur dioxide evolved, but also nitric acid and nitric oxide fumes. The arrangement is a convenient one, for, as the reaction proceeds and acid distils over into the U-tube, it is easy to allow part of this to flow into the beaker, and so to keep down the volume of liquid in the U-tube. Any gases not absorbed in the U-tube would be absorbed in the liquid in the beaker. One-half to 1 grm. of the rubber is cut up and placed in the flask with 20 to 30 c.c. of ordinary pure, strong nitric acid. The oxidation is conducted very gradually; the flask is gently heated to start the reaction, and then cooled so that not more than 0·5 inch of froth forms on the liquid. When the reaction is complete and the rubber dissolved the flask is again gently heated, so that the gases are slowly evolved and completely absorbed in the U-tube and beaker. The reaction is completed by heating more strongly until only 10 to 20 c.c. of colourless liquid remain in the flask. The contents of the flask are kept separate from those of the U-tube and beaker. The latter are united, taken to dryness, fused, dissolved in water, and precipitated with barium chloride after removal of nitric acid by repeated evaporation with hydrochloric acid. Working on these lines, sulphates were always found in the liquid contents of U-tube and beaker. Thus the vulcanised compound mentioned above gave 6·77 per cent. for sulphur contents of flask and 0·30 per cent. for contents of U-tube, making 7·07 per cent. in all.

In the next experiment the potash was replaced by water, as it was thought probable that some part of the sulphurous acid would be retained by the water. However, the contents of the U-tube on examination were found to give no precipitate with barium chloride, while the flask gave 6·85 per cent. of sulphur. This seems to negative the hypothesis of Bertrand and Potts. These experiments show that a varying loss of sulphur occurs, due to the formation of a volatile sulphur-containing substance during wet oxidation, this substance being soluble in potash, but not in water.

In the next experiments an unvulcanised mixing was employed, using potash in the U-tube, with results similar to those previously obtained. A number of experiments on soft rubber mixings, both vulcanised and unvulcanised, showed that the potash retained varying quantities of sulphur, usually from 0·2 to 0·5 per cent. calculated on the rubber. Potassium permanganate was next tried as an absorbent in the U-tube. As this substance reacts with nitrous fumes, a relatively large quantity had to be employed in order to preserve an excess until the oxidation was complete. It was found that the permanganate behaved similarly to the caustic potash, retaining most of the volatile sulphur compound given off during the oxidation of the rubber. The contents of the U-tube were heated with nitric acid to dissolve the precipitated oxide of manganese before proceeding to estimate the sulphuric acid formed. Thus, on oxidation of an unvulcanised mixing containing 8 per cent. of sulphur, there was obtained 7·01 per cent. of sulphur in the flask and 0·96 per cent. of sulphur by absorption with potash in the U-tube, making 7·97 per cent. in all; while in a parallel experiment the same figure, 7·01 per cent., was obtained for sulphur in the flask and 0·83 per cent. by absorption with permanganate in the U-tube, making a total of 7·84 per cent.

These two experiments instance the particularly heavy losses of sulphur which may take place when oxidising an unvulcanised mixing with nitric acid. It appeared of interest to determine at what stage the loss took place. An experiment was therefore made, leaving out the stopper of the flask during the first part of the reaction, and not inserting it until the rubber was dissolved, and then heating. Under these conditions, loss of sulphur undoubtedly took place, as is shown by the low figure for total sulphur—namely, 6·82 per cent.—of which 0·18 per cent. consisted of sulphur which had passed over into the U-tube and was absorbed by the potash during the subsequent heating of the flask, to complete the oxidation of the rubber. The actual amount of sulphur contained in this compound was 7·3 per cent., and two parallel experiments in which the stopper of the flask was not removed gave 7·28 per cent. Of this the amounts retained by the U-tube were 0·41 and 0·27 per cent. respectively.

A chance observation led the author to the discovery of one source from which loss of sulphur results. When oxidising the rubber in a flask, a very small quantity of a whitish, crystalline substance may occasionally be seen on the upper part of the neck of the flask and in the side-tube leading to the U-tube. This deposit is carried down into the U-tube by the condensed vapour. The substance was examined and found to be insoluble in water, but dissolved on heating with nitric acid, giving a solution containing a large quantity of sulphuric acid relative to the small quantity of material oxidised. From its appearance and behaviour this substance might be elemental sulphur, or possibly some complex substance of the nature of chamber crystals. Its occurrence is quite irregular, and as a general rule it is not formed. The author was unable to collect sufficient of the material to identify it with certainty. Whether sulphur or some substance rich in sulphur, it is possibly formed by the interaction of the gases in the flask, and deposited on the cool upper part of the neck and side-tube. Similarly, if the oxidation be carried out in a beaker, a small quantity of deposit may form on the clock-glass. It affords an explanation of the loss of sulphur during the oxidation process—at any rate, during the latter part of the same. If it consists of sulphur, it will, of course, be retained by potash in the U-tube of the apparatus above described, and will be oxidised during the fusion process. It will, however, remain undecomposed if the gases be passed into water, unless precautions are taken to acidify and boil with nitric acid before precipitating with barium chloride. As a fact, it is found that sulphuric acid is formed if these precautions are taken. Also, that the potash in the U-tube can be replaced by nitric acid, subsequently heating the nitric acid to oxidise the sulphur.

If the loss of sulphur be due to a mechanical action of some kind by which it passes off with the gaseous products of oxidation, it should be possible to devise some simpler form of apparatus for the estimation of sulphur in ordinary routine work than that employed in these experiments. For this purpose the author finds a flask with a form of Young's fractionating column about 1 foot long to work satisfactorily. This condenses all the vapours issuing from the oxidation-flask, and gives time for deposition of the sulphur. The author has also tried a long-necked flask such as a 300-c.c. Jena-Kjeldahl flask with a 6-inch condenser in the neck. So far the results appear satisfactory. A test on both methods is given in Table I.

Summary.—It has been shown that a varying loss of sulphur occurs during oxidation of rubber products with nitric acid, owing to the formation of volatile substances containing sulphur. Such volatile matter is retained for estimation as barium sulphate, by passing the gases evolved through a solution of potash, nitric acid, or even water, if this be subsequently oxidised with nitric acid. As an alternative, for general practice the volatile sulphur-containing products may be retained by providing the flask in which the oxidation is carried out with a suitable condenser. For this purpose there may be used either a 300-c.c. Kjeldahl flask with a 6-inch single surface condenser in the neck, or a 500-c.c. flask with a 12-inch Young's fractionating column with a ground-glass connection.

TABLE I.

Unvulcanised Compound.

(Containing 7·3 per cent. sulphur.)

		Sulphur, Per Cent.
Oxidation with nitric acid in a beaker covered with a clock-glass ...		7·02
Oxidation with nitric acid in a flask with Young's fractionating column		7·30
Oxidation with nitric acid in a 300-c.c. Kjeldahl flask with condenser in the neck		7·30
Oxidation as in last experiment		7·26
Oxidation in flask with nitric acid connected with U-tube, etc., also containing nitric acid.		
Sulphur from contents of flask	7·10	
,, ,, of U-tube	0·19	
	——	7·29

TABLE II.

Vulcanised Compound.

(Containing 7 per cent. of sulphur and vulcanised in thin sheet between plates.)

			Sulphur, Per Cent.
Oxidation with nitric acid in a beaker covered with a clock-glass ...			6·57
Oxidation in a flask with nitric acid connected to a U-tube containing water. The contents of U-tube subsequently oxidised with nitric acid.			
Sulphur from contents of flask		6·79	
,, ,, of U-tube		0·13	
			6·92
Separation of acetone soluble matter and oxidation of the extract and the extracted residue separately. In both cases oxidation with nitric acid in a flask connected to a U-tube containing water, and the contents subsequently oxidised with nitric acid.			
Acetone soluble matter:			
Sulphur from contents of flask		4·79	
,, ,, of U-tube		0·08	
			4·87
Extracted residue:			
Sulphur from contents of flask...	2·05		
,, ,, of U-tube	0·08		
		2·13	
			7·00
Oxidation in 300-c.c. Kjeldahl flask with condenser in the neck ...			6·96

DISCUSSION.

Dr. E. K. RIDEAL said that he had had a similar experience as to loss of sulphur. In a sample of shale suspected to contain small quantities of sulphur, an attempt had been made to detect the sulphur qualitatively by boiling with nitric acid and adding barium chloride. No sulphate, however, was obtained. But when a little of the same shale was warmed in a test-tube with fuming nitric acid and then tested for sulphates, a copious precipitate was obtained.

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THE COMPOSITION OF PALM-KERNEL OIL.

BY G. D. ELSDON, B.Sc., A.I.C.

INVESTIGATION CARRIED OUT UNDER THE SOCIETY'S ANALYTICAL INVESTIGATION SCHEME.

(*Read at the Meeting, December* 3, 1913.)

THE present paper deals with the results obtained from an examination of palm-kernel oil by the method of alcoholysis, this work being a continuation of that by the author on coconut oil (ANALYST, 1913, **39**, 8). In this former paper it was stated that the object of the work was to find, if possible, some differences between the constituents of these two oils which might be made use of in practice to detect, estimate, or differentiate these oils in mixtures.

Although cocoanut oil has been the subject of several investigations, very little seems to have been done in connection with palm-kernel oil; in fact, with the exception of the analytical figures and the statement that it differs but little from coconut oil, no references to this oil are to be found. This present communication is therefore, as far as the author is aware, the first that deals actually with its composition.

The Palm-Kernel Oil used.—Five kilograms of this oil were kindly placed at the disposal of the author by Messrs. Southall Bros. and Barclay of Birmingham. The oil used was said to be pure, and of a good average commercial quality, which statements are justified by the following analytical figures. For comparison the figures for coconut oil are also given:

	Palm-Kernel Oil.	Coconut Oil.
Zeiss butyro-refractometer (40° C.)	35·50	34·90
Iodine value (Wijs)	13·69	8·71
Saponification equivalent	25·11	25·84
Reichert-Meissl value	6·70	7·70
Polenske value	11·50	16·80
Kirschner value	1·80	2·50
Melting-point	25° C.	24° C.

The Process of Alcoholysis.—The full experimental details are given in the previous paper on coconut oil (*loc. cit.*). Using 500 grms. of the oil, the process of alcoholysis was complete in from twenty-five to thirty hours, and the resulting esters were separated in the usual manner. Four such treatments were carried out, so that finally a mixture of esters was obtained from 2 kgrms. of oil. These were then fractionated at 14 mm. pressure, when the following fractions were obtained:

Boiling-point.	Weight in grms.	Corresponding to—
63°– 76° C.	45·0	Methyl caproate
76°–100° C.	85·5	„ caprylate
100°–128° C.	108·5	caprate
128°–153° C.	1072·0	laurate
153°–182° C.	231·0	myristicate
182°–204° C.	172·0	palmitate
204°–216° C.	211·0	stearate and oleate

18·8 grms. of residue were obtained as a black semi-solid mass. This was found to consist of unchanged palm-kernel oil, some unsaponifiable matter which had become charred during the process, and a small quantity of methyl stearate. The whole was decolorised with animal charcoal and carefully crystallised and recrystallised from methyl alcohol, but no higher esters could be found. The melting-point of the product after recrystallisation was 37° C.

The various fractions were more or less completely purified (according to the amount of material available) and each ester identified. Each fraction was refractionated many times, but no further constituents were detected.

From the weights of the various fractions given above, a rough idea of the quantitative composition of palm-kernel oil can be gathered. It must be understood, however, that the appended figures, as was the case with coconut oil, are approximate only. For comparison the figures obtained for coconut oil are given alongside:

	Palm-Kernel Oil.	Coconut Oil.
Caproic acid	2	2
Caprylic acid	5	9
Capric acid	6	10
Lauric acid	55	45
Myristic acid	12	20
Palmitic acid	9	7
Stearic acid	7	5
Oleic acid	4	2

From the analytical point of view these figures are disappointing, for they merely indicate the differences in composition which are shown (and perhaps more clearly) by the usual analytical figures, whilst the latter are certainly easier to obtain. It will be seen that the chief differences in composition are a smaller percentage of lower fatty acids and a larger percentage of higher fatty acids in palm-kernel than in coconut oil. From the fact that coconut oil has both higher Reichert and Polenske values than palm-kernel oil, it would of course be expected that the percentage of lower fatty acids would be greater in coconut oil than in palm-kernel oil, whilst the greater iodine value of palm-kernel oil bears out the fact of the increased percentage of oleic acid. It follows therefore that the task of distinguishing between coconut oil and palm-kernel oil when present together in mixtures has not been made easier by the present work, since no distinguishing feature of either oils has been found; indeed, it is quite possible that some samples of coconut oil would be absolutely indistinguishable from some samples of palm-kernel oil, even in the pure condition.

The author has not tried the method of Burnett and Revis (ANALYST, 1913, **38**, 255), but it seems likely that this method may be found to afford useful information concerning the composition of mixtures of coconut oil and palm-kernel oil when average samples are concerned. However, such methods, which depend on the differences in percentages of some particular constituent, can never be really decisive on account of the natural variations among different samples of the same oil. In fact, at the present time the evidence seems to point to the fact that an exact method of estimating coconut oil and palm-kernel oil in mixtures, especially when other fats are present, is impossible.

As regards the merits of the method of alcoholysis, there is little to add to the opinions already expressed in the author's previous paper. Haller claimed that the process was a good qualitative one for certain oils, and a useful one for the preparation of the esters of the fatty acids. These claims are justified, but the process is not capable of being applied as a routine test, neither can the qualitative results be trusted to any great extent.

CITY OF BIRMINGHAM,
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DISCUSSION.

Mr. E. R. BOLTON remarked that the sample of palm-kernel oil examined by the author appeared, from its constants, to be somewhat abnormal. The Zeiss figure was very low—lower than would be accounted for by any ordinary proportion of free fatty acid. A more likely figure would be about 37°. The iodine value also was low, though just within the recognised limits, while the saponification value was very high. It seemed a pity that so much work should have been done on a sample which, although doubtless genuine, was abnormal to such an extent in regard to the ordinary constants. The figures given by the sample of coconut oil, on the other hand, were quite normal.

The PRESIDENT said that he was rather surprised at the author's remark that very little work had been done with regard to palm-kernel oil. His impression was that the author's work had been anticipated to some extent by Valenta.

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THE ESTIMATION OF STRYCHNINE IN THE PRESENCE OF QUININE.

BY CHARLES SIMMONDS, B.Sc.

(*Read at the Meeting, December* 3, 1913.)

THE accurate estimation of strychnine when mixed with quinine—for example, in such preparations as Easton's Syrup—has long been recognised as a somewhat difficult matter. Two processes are described in the new edition of Allen's "Commercial Organic Analysis" (vol. vi., pp. 461-462). In one of these the quinine is precipitated as tartrate, in the other as oxalate, from a solution of the mixed alkaloids, and the strychnine is determined in the residual liquid. A footnote, however (p. 462), suggests that the methods are not entirely satisfactory, the following results having been obtained by some observers:

	Strychnine Present. Grms. per 100 c.c.	Found.	
		Oxalate Method.	Tartrate Method.
Elixir	0·030	0·015	0·021
Syrup	0·020	0·0255	0·0258

In these circumstances it may be useful to describe a process worked out by the writer some years ago for the determination of the two alkaloids in the official "Syrup of Iron Phosphate with Quinine and Strychnine." The strychnine in this process is separated as the ferrocyanide from an acid solution of the mixed weighed alkaloids. The quinine may be either taken by difference or weighed separately, as desired.

Fifty c.c. of the syrup is a convenient quantity for analysis; it contains 0·5442 grm. of quinine and 0·0285 grm. of strychnine. These quantities of the alkaloids, one or both, in a volume of 50 c.c., were employed in the experiments to be described. The solution of potassium ferrocyanide used as a precipitant contained

$\frac{1}{100}$ of the grm.-molecular weight of the crystallised salt (4·2285 grms.) per 100 c.c.; but for analytical purposes this precise weight is unnecessary: a 4 per cent. solution suffices.

The principle of the method may be thus explained: In a neutral or only slightly acid solution of quinine sulphate a few drops of the ferrocyanide reagent will give a precipitate. If, however, the solution is strongly acid, a much larger quantity of ferrocyanide can be added before any quinine is thrown down. For example, to a solution of 0·544 grm. of quinine in 50 c.c. of dilute sulphuric acid containing 15 grms. of free H_2SO_4 about 11 c.c. of the ferrocyanide reagent may be added before an appreciable precipitate is obtained. (Less than this will give a precipitate on long standing.) Now, even 1 c.c. of the ferrocyanide reagent contains enough of the salt to precipitate 0·0285 grm. of strychnine from a neutral or slightly acid solution. With some excess of ferrocyanide the strychnine can also be completely thrown down from a solution that is strongly acid.

Experiment 1: *Strychnine without Quinine.*—0·0285 grm. in 50 c.c. of 20 per cent. sulphuric acid (20 grms. H_2SO_4 per 100 c.c.):

Volume of Ferrocyanide.	Strychnine recovered.
3 c.c.	0·0284 grm.
5 „	0·0284 „

Hence, if there were no question of the co-precipitation of quinine, the strychnine might be separated from a strongly acid solution of the two alkaloids by using sufficient ferrocyanide, but avoiding large excess. Some co-precipitation of quinine, however, always occurs. The quantity is small if the excess of ferrocyanide is slight, and increases with the excess.

Experiment 2: *Strychnine with Quinine.*—0·0285 grm. strychnine and 0·5442 grm. quinine in 50 c.c. of 20 per cent. sulphuric acid:

	Volume of Ferrocyanide Solution used.			
	1 c.c.	2 c.c.	3 c.c.	9 c.c.*
Alkaloids obtained	0·0296	0·0322	0·0356	0·0434 grm.
Excess over strychnine ...	0·0011	0·0037	0·0071	0·0149 „

This co-precipitated quinine, however, can be eliminated, and the strychnine recovered practically pure, by a reprecipitation under the original conditions as regards acid and ferrocyanide solution. Thus the foregoing four quantities gave on reprecipitation the following weights of strychnine:

0·0270 grm.; 0·0272 grm.; 0·0278 grm.; 0·0282 grm.

That is, substantially the whole of the strychnine is recovered in the last case, and there is but little loss in the first three experiments.

The chief conditions required are therefore—(1) Sufficient concentration of acid to prevent the co-precipitation of an unduly large quantity of quinine; (2) sufficient ferrocyanide to insure the complete precipitation of the strychnine, but not so much as to give an unnecessarily large quantity of co-precipitated quinine; and (3) reprecipitation of the impure strychnine ferrocyanide given by the first operation.

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* Twenty-five per cent. acid used in this experiment.

From experiments made with varying quantities of acid and potassium ferrocyanide it was found that good results were obtained by using 20 per cent. acid and 5 c.c. of the potassium ferrocyanide solution. The process as finally employed was as follows: The mixed alkaloids from 50 c.c. of the syrup are obtained in the usual way by extraction with chloroform and ether after adding citric acid and ammonia. These alkaloids are weighed, and dissolved in 50 c.c. of 10 per cent. sulphuric acid. Five c.c. of potassium ferrocyanide solution (4 per cent.) are run in from a burette drop by drop, stirring well, and the mixture set aside for a few hours. The precipitate is filtered through a small filter (7 cm.), and washed lightly three times with about 3 c.c. of 5 per cent. sulphuric acid. With the aid of about 10 c.c. of dilute ammonia solution (10 per cent.) and a fine jet of water the precipitate is washed into a small separator, and extracted three times with chloroform, using 15 c.c., 10 c.c., and 5 c.c. The chloroform solutions are collected in another separator, and the alkaloids extracted from them with 50 c.c. of the 20 per cent. sulphuric acid, using 30 c.c., 10 c.c., and 10 c.c.; then the precipitation and other operations are repeated as before until the chloroform extracts are again obtained. The chloroform is evaporated carefully, a little alcohol being added towards the end to prevent sputtering, and the residue of strychnine weighed after drying it for an hour or so at 100° C.

If it is desired to weigh the quinine separately, the two filtrates are united and made up to a convenient volume (250 c.c.), the quinine being then estimated in an aliquot portion (50 c.c.) by precipitation with ammonia and extraction with chloroform as usual.

Duplicate experiments gave—

Strychnine	0·0283	and 0·0286	instead of 0·0285	grm.
Quinine...	0·5450	„ 0·5450	„ 0·5442	„

Though devised specially for the proportions of alkaloids in the official syrup, the method can be used for any mixture of strychnine and quinine. Thus, from a solution containing 0·5 grm. quinine and 0·005 grm. strychnine, the quantity of the latter recovered was 0·0048 grm. The weight of alkaloids taken, however, must be such that not much more than 0·1 grm. of strychnine is present in the 50 c.c. of acid; at somewhat higher concentrations strychnine sulphate begins to separate out.

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

Detection of Beef-Fat in Lard. A. Bömer, R. Limprich, R. Kronig, and J. Kuhlmann. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1913, **26**, 559-618.)—The method proposed depends on the difference shown between the melting-points of the more insoluble glycerides of beef-fat and lard and of the respective fatty acids separated from these glycerides. For instance, the glycerides separated by crystallisation from lard have a melting-point of 68·5° C., and the fatty acids obtained from the glycerides melt at 63·3° C., a difference of 5·2° C.; in some lards this difference may amount to 6·9° C. In the case of beef and mutton fats the difference varies from 0·1° to 2·6° C. The glycerides are obtained by mixing 50 grms. of molten and filtered fat with 50 c.c. of ether, allowing the mixture to stand at 15° C. for one hour, and then collecting the crystalline mass on a filter. The mass is pressed slightly to remove the liquid portion as much as possible, and is then dissolved in a further quantity of 50 c.c. of ether and allowed to crystallise for one hour. The crystalline mass is again collected on a filter, pressed, allowed to dry, and the melting-point is determined. If the crystals melt below 61° C., as happens in a few cases, they must be recrystallised from ether. Very liquid fats may not yield crystals when thus treated, and in this case a mixture of ether and alcohol may be used as the solvent. A portion of the crystalline mass is then saponified, the fatty acids are separated, and their melting-point is determined. It is essential that the melting-point be determined on the crystals themselves in the case of the glycerides, but the fatty acids may be melted and allowed to solidify previous to the determination. With glycerides melting between 60° and 61° C., the presence of beef-fat is indicated when the difference between the melting-points of the glycerides and the fatty acids is less than 5·0° C., or when the difference is less than 3° C. in the case of glycerides melting between 65° and 68·5° C. The presence of about 10 per cent. of beef-fat in lard may be detected by the method.

W. P. S.

Detection of Phytosterol in Animal Fats. M. Fritzsche. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1913, **26**, 644-648.)—The following procedure is recommended for separating phytosterol or cholesterol from fats by means of digitonine (ANALYST, 1910, **35**, 256; 1913, **38**, 458): 50 grms. of the filtered fat are mixed with 20 c.c. of a 1 per cent. alcoholic digitonine solution and heated to 70° C. for five minutes, the mixture being stirred continuously. When the fat under examination is fluid or semifluid, the mixture is filtered directly after the heating, and the insoluble residue is washed six times with ether, using 5 c.c. for each washing. In the case of solid fats, it is advisable to add 20 c.c. of chloroform before filtering the mixture; the residue is then washed twice with warm chloroform and six times with ether. The fat-free residue on the filter is dried at 40° C., then removed from the filter and boiled with 2 c.c. of acetic anhydride for five minutes; the hot solution is now filtered through cotton-wool, the latter is washed with 1 c.c. of hot alcohol, and the filtrate and washing are evaporated to dryness in a small basin. The residue is dissolved in 1 c.c. of boiling alcohol, allowed to crystallise, the crystals are dried on a porous plate, then heated for ten minutes at 80° C., and the melting-point is determined. This constant is again determined after recrystallisation. The following were the melting-points of the acetates (second crystallisation) obtained from various fats: Beef-fat, 113·4° to 113·7° C.; tallow, 114·0° C.; oleomargarine, 113·9° to 114·5° C.; lard, 114·0° to 114·5° C.; butter-fat, 114·4° C. The presence of paraffin in the fat does not have any influence on the melting-point of the acetates.

W. P. S.

Analysis of Coffee (Estimation of Roasted Cereals in Coffee Mixtures). I. Rozsenyi. (*Chem. Zeit.*, 1913, **37**, 1482-1484.)—Estimations of the total ash, soluble ash, and alkalinity of the ash are recommended as affording data for calculating the quantity of roasted cereal in a coffee mixture. Roasted coffee yields from 4·06 to 4·80 per cent. of ash and from 3·36 to 4·08 per cent. of soluble ash. Each grm. of the ash requires from 11·50 to 12·18 c.c. of $\frac{N}{1}$ acid for neutralisation. Roasted barley, wheat, and rye give from 1·76 to 1·85 per cent. of ash, and each grm. of the latter requires from 0·51 to 0·90 c.c. of $\frac{N}{1}$ acid for neutralisation. From 79 to 88 per cent. of the mineral matter present in coffee passes into solution when the coffee is extracted with water. W. P. S.

Analysis of Maple Products, II. J. F. Snell and J. M. Scott. (*J. Ind. and Eng. Chem.*, 1913, 5, 993-997.)—The paper is an extension of the work of Snell (Analyst, 1913, **38**, 499) on the electrical conductivity method for the detection of adulteration in maple syrup, and consists of a comparison of this method with the principal ones in vogue. The delicacy of any such method depends on the limits of natural variation of the value in question in genuine syrups, and the rate at which this value falls off as the proportion of genuine syrup is decreased. Three samples of maple syrup were dealt with and diluted with varying quantities of cane-sugar syrup, the rates at which the conductivity values, ash data, and various lead values fell off on progressive dilution being determined. The range of variation of these data in genuine syrups was also studied with reference to the work of Bryan and McGill. McGill's (*Lab. Inland Rev. Dept., Ottawa*, Bull. 1911, **228**, 5) lead value showed the most rapid falling off, and the conductivity value the narrowest range. Winton and Kreider's (Analyst, 1906, **31**, 411) lead method was found to give better agreement for duplicate determinations than McGill's method. In the latter method no material difference was noticeable whether the precipitate was washed with water at 80° and 100° C., and practically identical results were obtained when using 100 c.c. and 150 c.c. of washing-water; but the results were higher when 50 c.c. of water was used. The use of cane-sugar syrup instead of acetic acid as recommended by Bryan in the Winton method is suggested. A large number of results is tabulated in full, seven ash determinations (total, soluble, alkalinity of soluble and insoluble ash, etc.), four different lead values, and a conductivity determination being recorded for the three original maple syrups when diluted with cane syrup from 10 to 90 per cent., values being given for each 10 per cent. dilution. H. F. E. H.

Constants for the Double Polarisation [Clerget-Herzfeld] Method of Determining Cane Sugar. L. G. Langguth Steuerwald. (*Archief Suikerind. Nederl.-Indië*, 1913, **21**, 1383-1392.)—At the last meeting of the International Commission for Uniform Methods of Sugar Analysis, held at New York on September 12, 1912, it was pointed out that the table of constants constructed by Herzfeld for use in the double polarisation method of determining cane-sugar, to take account of the change of rotation at different concentrations, was apparently inaccurate, giving somewhat too high results, and at the suggestion of Herzfeld a committee was appointed to investigate the matter. Working in Java, with every possible precaution to insure accuracy, the author has drawn up the following amended table of constants, by which it will be noted that, in place of the generally used Herzfeld value of 142·66 for a concentration of 13 grms. of sugar per 100 c.c., an appreciably higher one—namely, 143·05—is found. Hence for the concentration mentioned, the formula for the calculation of the percentage of cane sugar by the double polarisation method will be: $\frac{D+I}{143{\cdot}05-\frac{1}{2}t}$, D and I being the direct and inversion readings, and t the temperature of observation.

Grms. of Cane Sugar per 100 c.c.	Constant for 0° C.	Grms. of Cane Sugar per 100 c.c.	Constant for 0° C.
1	142·19	11	142·91
2	142·26	12	142·98
3	142·34	13	143·05
4	142·41	14	143·12
5	142·48	15	143·20
6	142·55	16	143·27
7	142·62	17	143·34
8	142·69	18	143·41
9	142·77	19	143·48
10	142·84	20	143·55

J. P. O.

Estimation of Saccharin in Foods. H. Durand. (*J. Ind. and Eng. Chem.*, 1913, **5**, 987-989.)—Five methods were investigated, and the author prefers the method of Schmidt, which depends upon the conversion of the saccharin into salicylic acid. The latter acid, if present, may be removed by the addition of bromine. H. F. E. H.

Detection of Sesame Oil in Margarine containing Colouring Substances. W. Arnold. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1913, **26**, 655.)—Margarines sometimes contain added colouring substances which yield a red coloration when the sample is treated with hydrochloric acid, but this coloration does not interfere with Baudouin's test for sesame oil if a small quantity of stannous chloride is added to the hydrochloric acid; about 0·1 c.c. of stannous chloride solution per 100 c.c. of the acid is sufficient for the purpose. The fat is dissolved in petroleum spirit and shaken with hydrochloric acid containing stannous chloride, the mixture being then heated by placing the test-tube for a short time in boiling water. When the red coloration has disappeared, the furfural solution is added, and the test carried out in the usual way.

W. P. S.

Sulphur Dioxide in Gelatin. P. Poetschke. (*J. Ind. and Eng. Chem.*, 1913, **5**, 980-987.)—When the sulphur dioxide liberated from gelatin by distillation with phosphoric acid in an atmosphere of carbon dioxide is estimated by the iodine and thiosulphate method, an error is introduced owing to the presence in the distillate of reducing substances other than sulphurous acid; the gravimetric method in which the resulting sulphuric acid is estimated as barium sulphate is therefore better. It was not found necessary to use steam for distillation; careful experiments made by this method and with direct boiling failed to show any discrepancies. Prolonged heating in the presence of phosphoric acid with a resulting increase in concentration and acidity does not cause any dissociation of the organic sulphur of the gelatin with liberation of volatile sulphur compounds. Gelatin readily absorbs traces of sulphur dioxide from the atmosphere, and there is no doubt that it is in this manner that samples made without the direct employment of this chemical as a bleaching agent have acquired the impurity. Of the samples from all sources examined in 1912, 43 per cent. contained from 100 to 500 parts of sulphur dioxide per million, 3·3 per cent. over 1,000 parts per million, and 48 per cent. under 100 parts per million, 20 per cent. being under 10 parts per million, and of these last a number contained hydrogen peroxide, evidently added to destroy sulphur dioxide. H. F. E. H.

Estimation of Total Sulphurous Acid in Wine. L. Levy. (*Ann. Falsif.*, 1913, **6**, 595.)—The process depends on the oxidation of sulphurous acid by hydrogen peroxide. Fifty c.c. of the wine are mixed in a stoppered flask with 2 grms. of barium peroxide, and the mixture is allowed to stand for about 16 hours. It is then acidified with hydrochloric acid, ammonium chloride added, the solution boiled, and the barium sulphate is collected on a filter, washed with water containing a small quantity of ammonium chloride, dried, ignited, and weighed. The quantity of sulphates already existing in the wine is estimated on a separate portion of the sample and the difference between the results of the two estimations is calculated into sulphur dioxide. W. P. S.

Estimation of Glycerol in Wine. S. Rothenfusser. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1913, **26**, 535-544.)—Further details are given concerning the method previously described by the author (ANALYST, 1912, **37**, 260), the new matter dealing mainly with the quantities of reagents required to remove oxidisable substances other than glycerol from the wine. In the case of wines containing not more than 1 per cent. of sugars, 50 c.c. of the sample are treated with 30 c.c. of 20 per cent. sodium carbonate solution and 5 grms. of crystallised stannous chloride, the mixture is diluted to 250 c.c., and filtered. Two hundred c.c. of the filtrate are then mixed with a freshly prepared solution consisting of 40 c.c. of basic lead acetate solution and 20 c.c. of 10 per cent. ammonia, the mixture is diluted with 10 per cent. ammonia to 300 c.c., and filtered. To 200 c.c. of the filtrate are added 5 c.c. of 10 per cent. sodium phosphate solution and 6 grms. of anhydrous sodium carbonate, and the mixture is evaporated to about 60 c.c. The solution and precipitate are now rinsed into a flask, diluted to 100 c.c., cooled to 25° C., and 10 grms. of potassium carbonate and 2 grms. of potassium permanganate are added. After forty-five minutes the excess of permanganate is destroyed by the addition of hydrogen peroxide, the solution is diluted to 250 c.c., filtered, and the oxalic acid is estimated in the filtrate as described (*loc. cit.*). The calcium oxalate should be precipitated from an acetic acid solution.

With wines such as port and sherry, containing about 6 per cent. of sugars, 5 c.c. are mixed with 15 c.c. of sodium carbonate solution and 2·5 grms. of stannous chloride, diluted to 250 c.c., and filtered. Two hundred and twenty c.c. of the filtrate are then treated with an excess of ammoniacal lead acetate solution (equal volumes of basic lead acetate solution and 10 per cent. ammonia), about 20 c.c. of 10 per cent. ammonium carbonate solution are added, and the whole is diluted with 10 per cent. ammonia to 500 c.c. Before the addition of the ammonium carbonate a small volume of the mixture is filtered and the filtrate tested with ammonium sulphide to make certain that an excess of lead is present. The mixture is then filtered, 250 c.c. of the filtrate (which should not yield the faintest reduction when boiled with Fehling's solution) are evaporated with the addition of 5 c.c. of 10 per cent. sodium phosphate solution and 6 grms. of anhydrous sodium carbonate, and the estimation is proceeded with as described above.

In the case of wines containing about 20 per cent. of sugars, 50 c.c. are treated with 15 c.c. of sodium carbonate solution and 2·5 grms. of stannous chloride, 100 c.c. of water are added, the mixture is heated to 50° C., then diluted to 250 c.c., and filtered. Two hundred and twenty c.c. of the filtrate are treated with an excess of a mixture consisting of 180 c.c. of basic lead acetate solution and 70 c.c. of 10 per cent. ammonia, and the estimation is then carried out as described.

When large quantities of sucrose are present in the wine under examination this sugar should be inverted by heating the wine with hydrochloric acid before proceeding with the estimation of the glycerol. W. P. S.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

The Hydrolysis of Lævulosans and its Application in Plant Analysis, II. and III. P. de Vilmorin and F. Levallois. (*Bull. Soc. Chim.*, 1913, **13**, 1060-1063; 1063-1068.)—In a previous paper (Analyst, 1913, **38**, 429) the authors showed that 1.3.5 sulphosalicylic acid gave the most constant and readily controlled results in the hydrolysis of inulin, but that the reaction reached its maximum limit with the formation of about 92 per cent. of reducing sugars, expressed as invert sugar on the weight of the inulin. Discussing the causes of this deficiency, the authors have ascertained that it was not due to any peculiarity in the inulin employed, since samples of inulin of other origins all yielded practically the same amount of reducing sugar. Neither can it be attributed in any large degree to the formation of a non-reducing by-product of the nature of a reversion-dextrin. When lævulose was heated with acids under the conditions and at the dilutions adopted in their method of hydrolysis (*loc. cit.*), the loss in cupric reducing power was not more than 1 per cent. Inulin dextrins have been observed, but only in the hydrolysis of highly concentrated solutions; the authors, however, obtain the same results with 10 per cent. solutions of inulin as with 1 per cent. Nevertheless, there is a certain amount of evidence of some secondary reaction, because the hydrolysis follows the law of a monomolecular reaction, but with a velocity coefficient which gradually decreases as the reaction progresses. The authors also discuss the method of calculating the cupric reducing values in terms of "invert sugar" in view of the fact that the coefficient varies according to the manner in which the inversion of the sucrose is carried out. They made a mixture of equal parts of purified dextrose and lævulose; estimating the cupric reducing values by Bertrand's method, they found the value of the mixture always slightly higher than the mean of the cupric reducing values of the two sugars separately. Thus, with 50 mgrms. of sugar they found for pure dextrose 99·1 mgrms. of copper; for pure lævulose, 92·6; for the mixture, 96·5; calculated, 95·9. But when the mixture of sugars was heated with acids under various conditions commonly employed for the inversion of sucrose, a very distinct increase in cupric reducing values was recorded, and the copper equivalent for 50 mgrms. of sugar became 101·3 for sugar heated by Bertrand's inversion method and 99·8 for that heated by Clerget's method. The authors favour the adoption of a method proposed by Pellet for the estimation of cupric reducing values by heating with Fehling's solution on the water-bath at a temperature of only 90° C. Many of the abnormal features of the ordinary method are thereby eliminated, and the factor becomes almost constant for different quantities of sugar. By this method 50 mgrms. of dextrose reduced 94·5 of copper, and the same weight of lævulose reduced 85·5, a mixture of the two yielded the amount calculated from the mean of the components, and its value agreed with that of invert sugar prepared from sucrose by Bertrand's and Clerget's methods. Finally, the authors confirm the fact that, whether the products of hydrolysis of inulin be calculated as invert sugar, lævulose, or a mixture of 11 parts of lævulose to 1 of dextrose, the hydrolysis reaches a limit when 90·5 to 91 per cent. of the theoretical quantity of sugar is produced. J. F. B.

Treatment of Sinews, particularly by the Application of Ruthenium Red and the Mallory Connective Tissue Stain. M. Heidenhain. (*Zeitsch. wiss. Mikroskop.*, 1913, **30**, 161-167; through *Chem. Zentralbl.*, 1913, II., 2163.)—Ruthenium red, a compound of ruthenium oxychloride and ammonia, should be kept completely protected from the air; it quickly stains dried sinews an intense red. The stained preparations may be preserved for a long time in 10 per cent. alcohol, but ultimately fade to a pale brown. The ground-substance of cartilage is also stained a deep purple-red by ruthenium red, and the so-called cartilage residues of the embryonic spongiosa are intensely stained. The process of fixing and staining sinew-fibrillæ and sinew-cells by means of iron-hæmatoxylin is discussed, and the morphologically and chemically different mode of formation of muscles and sinews is set forth to show that a direct transition between these matters does not take place. For the staining of muscles and sinews a modification of Mallory's stain is employed. Instead of the treatment with acid fuchsin S, the author prefers to stain with borax-carmine in the mass, or to stain the sections with carmine-alum or azocarmine B. The original staining solution is diluted with four times its volume of water, and the sections allowed to remain twenty to thirty minutes in the liquid. J. F. B.

Estimation of the Lime Requirement of Soils. J. A. Bizzel and T. L. Lyon. (*J. Ind. and Eng. Chem.*, 1913, **5**, 1011-1012.)—Since Veitch's method (*J. Amer. Chem. Soc.*, 1902, **24**, 1120) requires at least six independent experiments, and gives much trouble with certain types of soil, the authors have examined a much older method due to Albert (*Zeitsch. angew. Chem.*, 1888, **1**, 533). As originally described, this method yielded results which differed widely from those obtained by Veitch's method, and which field results showed were much too low. By slightly modifying Albert's method, however, the authors obtain results in close agreement with those obtained by Veitch's method, which, if not entirely satisfactory, has been the best method so far available. The new method consists in mixing 25 grms. of the soil with 50 c.c. boiled distilled water and 50 c.c. $\frac{N}{10}$ barium hydroxide, heating the mixture in a boiling-water bath for an hour, with occasional shaking, addition of 150 c.c. distilled water and 5 grms. ammonium chloride, and distillation and estimation of the ammonia set free by the excess of barium hydroxide, over and above that absorbed by the soil. As some ammonium chloride is decomposed on boiling with water and soil alone, a blank experiment is made without barium hydroxide. The ammonia obtained in this blank experiment is deducted from that obtained in the experiment with barium hydroxide. The amount of barium hydroxide unabsorbed is thus given, and the portion absorbed by the soil is calculated to its lime-equivalent. G. C. J.

Micro-Estimation of Sugar in Blood. J. Bang. (*Biochem. Zeitsch.*, 1913, **57**, 300-313; see ANALYST, 1913, **38**, 218.)—The author recommends reducing the volume of copper solution to 1 c.c., and using 13 c.c. (6·5 c.c. + 6·5 c.c.) of a solution prepared by adding 1·5 c.c. 25 per cent. hydrochloric acid + 640 c.c. water to 1,360 c.c. of a saturated potassium chloride solution for the coagulation of proteins instead of acetic acid. The strength of the iodine solution may be reduced to $\frac{N}{200}$, but it is necessary to make a blank estimation and apply a correction. The author recommends the application of his method to the estimation of sugar in urine.

E. W.

Estimation of Sugar in Blood. W. Griesbach and H. Strassner. (*Zeitsch. Physiol. Chem.*, 1913, **88**, 199-210.)—The authors state that the cupric reducing power and specific rotation are due almost exclusively to dextrose; blood-filtrates after fermentation do not reduce, and are optically inactive even when abnormally large quantities of yeast have been used. Sugar estimations by polarisation and copper reduction give concordant results. The methods of Maquenne and Bertrand give identical results, whilst Tachau's (*D. Arch. f. kl. Med.*, 1911, **102**, 597) method gives good results for human blood, but somewhat low results for dog's blood. For clinical purposes, Bang's micro method was found to be as good as other methods. E. W.

ORGANIC ANALYSIS.

Estimation of Loading in Caoutchouc Mixtures according to the Standards of the Verband deutscher Elektrotechniker. F. Jacobsohn. (*Gummi-Zeit.*, 1913, **27**, 1906-1907; through *Chem. Zentralbl.*, 1913, II., 2173-2174.) A method for the analysis of electrical wire insulations has been agreed upon in connection with the standard specifications. A weighed quantity of the rubber is dissolved by long heating with solvents of high boiling-point, and the undissolved portion is separated either by filtration or centrifuging. In addition, the organic loading materials soluble in acetone are estimated, and the caoutchouc is calculated by difference. As solvents, a choice is given between petroleum of b.-pt. 230° to 260° C., and in case this is not sufficiently powerful, camphor oil and paraffin oil. The author has carried out comparative estimations with a standard mixture containing 33·3 per cent. of caoutchouc. He found that paraffin oil has an insufficient solvent power and yields caoutchouc values which are too low, whereas camphor oil, when used alone, gives too high results for the caoutchouc. The values found ranged between 43·5 and 32·13 per cent. With a suitable combination of the two solvents correct results were obtained.

Commenting on the above communication, P. Alexander (*Gummi-Zeit.*, 1913, **27**, 2045; through *Chem. Zentralbl.*, 1913, II., 2174) states that the only conclusion to be drawn is that the normal method of the Verband deutscher Elektrotechniker does not afford a sufficiently accurate estimation of the mineral loading matters, and consequently of the percentage of caoutchouc. This view has frequently been expressed by the author since the normal standards were fixed. In a later reply (*Gummi-Zeit.*, 1913, **27**, 2116), Jacobsohn agrees with this view. J. F. B.

Application of Diphenylcarbohydrazide (Diphenylcarbazide) as Indicator in Titration of Iron with Dichromate. L. Brandt. (*Zeitsch. anal. Chem.*, 1914, **53**, 1-20; *cf.* Barneby and Wilson, ANALYST, 1913, **38**, 167.)—The author, who first proposed the use of this indicator, agrees with Barneby and Wilson that it is itself a reducing agent, but, instead of making a blank test as they do, he makes the method an empirical one, standardising the dichromate on 0·7 grm. of ferric oxide, and taking always for assay a quantity of ore such that from 0·6 to 0·8 grm. ferric oxide is present. The method is available even in presence of as much trivalent arsenic as corresponds to 2 c.c. of $\frac{N}{10}$ solution—that is to say, with iron ores containing rather more than 0·5 per cent. of arsenic. Larger quantities of trivalent arsenic interfere, and must be oxidised before the iron is reduced with stannous chloride, which, as is known (Chemiker-Kommission des Vereins deutscher Eisenhüttenleute, *Stahl u. Eisen*, 1908, **28**, 508), does not reduce arsenic acid under the conditions of the usual reduction of iron as a preliminary step towards its estimation by dichromate or permanganate. The fact that small amounts of trivalent arsenic exercise a scarcely measurable influence on the results is shown to depend on the use of manganese sulphate and phosphoric acid. Copper must be absent, as its presence introduces irregularities into the results for which no correction is possible.

G. C. J.

Colorimetric Estimation of Histidine. M. Weisz and N. Ssobolew. (*Biochem. Zeitsch.*, 1913, **58**, 119-130.)—Histidine can be estimated by diluting a solution of it until the colour produced on adding diazobenzenesulphonic acid is equal to that produced in a standard histidine solution (1 part histidine hydrochloride in 10,000 parts water) by the same reagent. The diazobenzenesulphonic acid is freshly prepared by mixing 1 part of sulphanilic acid solution (4 grms. sulphanilic acid, 40 c.c. concentrated hydrochloric acid, 400 c.c. water) with 2 parts of a 0·5 per cent. sodium nitrite solution. The liquid to be tested (10 c.c.) is treated with 1·5 c.c. of the freshly prepared diazobenzenesulphonic acid solution and the mixture made alkaline by adding 3 c.c. of a 10 per cent. solution of anhydrous sodium carbonate. Since concentrated histidine solutions do not give this colour reaction it is always necessary to test whether the colour develops on dilution. If other substances are present which react with diazobenzenesulphonic acid, the quantity of this reagent used must be increased so as to yield the maximum coloration.

E. W.

Modification of Halphen's Test for Cotton-Seed Oil. Utz. (*Chem. Rev. Fett Ind.*, 1913, **20**, 291-295.)—The substitution of pentachlorethane for carbon bisulphide in this test enables the mixture to be heated to a higher temperature and renders the reaction more rapid, intense, and sensitive, than has hitherto been possible. The mixture of 5 c.c. of oil, 5 c.c. of amyl alcohol, and 5 c.c. of a 1 per cent. solution of sulphur in pentachlorethane (b.-pt. 159° C.) is heated over an iron plate until the liquid gently boils. In this way the presence of 1 per cent. of cotton-seed oil in other oils could be detected with certainty. The same results were obtained without the addition of amyl alcohol at the higher temperature of the test, but at the temperature of boiling water the reaction took place very slowly and was promoted by the amyl alcohol.

C. A. M.

Gravimetric Estimation of the Oxygen Absorption of Linseed Oil during Drying. Krumbhaar. (*Chem. Rev. Fett Ind.*, 1913, **20**, 290-291.)—An exact method of estimating the oxygen absorbed during the drying of linseed and other oils is described. The weighed quantity of oil is mixed with 0·6 per cent. of cobalt resinate and spread over filter-paper, which is placed in a U-tube immersed in water at 30° C. throughout the absorption. A steady current of dry air (free from carbon dioxide) is drawn through this tube, and the volatile products leaving the tube are passed, first through a strongly heated tube of copper oxide (to complete their decomposition into carbon dioxide and water), and then through tared absorption tubes containing calcium chloride and soda-lime respectively. After every two hours the air in the apparatus is replaced by nitrogen, the taps closed, and the increase in weight of the tube containing the oil and of the two absorption tubes determined until the weight of the oil tube becomes constant. The sum of the increase of weight in the oil tube, of the hydrogen absorbed by the calcium chloride tube, and of the carbon absorbed by the soda-lime tube, gives the amount of oxygen taken up by the oil. For example, in one experiment 0·743 grm. of linseed oil showed an increase in weight of 0·128 grm. in eighteen hours, whilst the amounts of volatile hydrogen and carbon were 0·009 and 0·016 grm. respectively, giving a total of 0·153 grm. or 20·6 per cent. of oxygen absorbed. It is suggested that this method may afford a means of distinguishing between different drying oils such as soya bean, tung, and linseed oils.

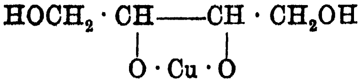
C. A. M.

Montanin and Montana Waxes. H. Ryan and T. Dillon. (*Scient. Proc. Roy. Dublin Soc.*, **12,** 202-209; through *Chem. Zentralbl.*, 1913, II., 2048-2049.)—Montana wax from Irish turf is a yellow crystalline body melting at 76° C. It is sparingly soluble in cold alcohol, ether, chloroform, and petroleum spirit, but dissolves readily in the hot solvents. A sample, consisting of 53 per cent. of acid and 47 per cent. of unsaponifiable matter, gave the following values: acid value, 73·3; saponification value, 73·9; and iodine value, 16·0. It contained no resin or cholesterol. The acid, which consisted almost entirely of montanic acid, $C_{28}H_{56}O_2$, could be crystallised from alcohol in crystals melting at 83° C. The unsaponifiable matter was crystallised from benzene in fine needles melting at 58° to 59° C. It had a sp. gr. of 0·92 and was composed of 83·56 per cent. of carbon, 14·0 per cent. of hydrogen, and 2·44 per cent. of oxygen. It contained neither primary nor secondary alcohols. The wax was thus materially different from the peat wax of Zaloziecki and Hausmann (ANALYST, 1907, **32,** 336). Another wax termed montanin wax (m.-pt. 95° to 97° C.) differed only from montana wax in the fact that a part of the montanic acid was present in the form of its sodium salt, so that the wax was harder. Wax from Irish lignite gave values similar to those of montana wax. C. A. M.

Estimation of Nitrogen in Nitro-Compounds by Kjeldahl's Method. A. Eckert. (*Monatsh.*, 1913, **34,** 1957.)—This method depends upon the reduction of the nitro-compound by means of sulphur. A mixture of 0·2 to 0·5 grm. of the substance with 4 grms. sulphur is warmed in a Kjeldahl flask on the water-bath, with 15 to 20 c.c. of a 40 per cent. fuming sulphuric acid ("oleum"), for one hour. The reduction is then complete, and the estimation is finished as usual. Analyses of nitrobenzene, bromonitrobenzene, and similar compounds showed satisfactory results.

O. E. M.

Estimation of Mannitol. J. Smit. (*Chem. Weekblad*, 1913, **10,** 894-907; through *Chem. Zentralbl.*, 1913, II., 2169). Wagenaar's method for the estimation of glycerol (*Chem. Zentralbl.*, 1911, II., 103) may be applied without modification for the estimation of mannitol. The quantity of mannitol corresponding to the number of c.c. of thiosulphate used is ascertained from an empirical table or curve. The following substances interfere with the estimation since they consume small quantities of thiosulphate: ammonium compounds, which may be expelled by boiling with alkali; amino-acids with several amino groups, which may be precipitated with phosphotungstic acid or converted into hydroxy-acids by nitrous acid; sugar, which may be removed by fermentation or decomposition; hydroxy-acids, which are precipitated as lead or calcium salts; higher alcohols; and glycerol, which is dissolved with alcohol-ether. The estimation in presence of sorbitol, dulcitol, etc., is tedious. In this case the estimation is carried out by combining the mannitol with benzaldehyde or acetal. The following structural formula of the copper compound of mannitol is given:



$$\begin{array}{c} HOCH_2\cdot\underset{|}{CH}\text{——}\underset{|}{CH}\cdot CH_2OH \\ O\cdot Cu\cdot O \end{array}$$

J. F. B.

Examination of Nitrocelluloses with the Polarising Microscope. H. Ambronn. (*Kolloid-Zeitsch.*, 1913, **13**, 200-207; through *Chem. Zentralbl.*, 1913, II., 2175-2176.)—A perfectly definite relationship exists between the optical properties of nitrocellulose fibres and the percentage of nitrogen, so that higher and lower degrees of nitration may be detected under the microscope, and the uniformity or otherwise of the sample may be estimated. Purified cotton is strongly doubly refractive and shows between crossed nicols various interference colours of the first order, varying according to the thickness of the fibres, but principally bright yellow. The fibres are positive in double refraction relatively to the direction of their length. Collodion cottons show colours of the first order, chiefly lavender to grey-blue; they are positive in double refraction, but their anisotropism is distinctly weaker than with pure cotton. Highly nitrated gun-cotton is coloured blue in polarised light, and the double refraction is weakly negative in relation to the longitudinal direction of the fibre. Thus there is a change in sign of the double refraction which passes through zero at a degree of nitration corresponding to about 11·8 per cent. of nitrogen; this change takes place gradually with increasing percentage of nitrogen. In a number of samples it was established that the single fibres were uniformly nitrated throughout their length, but in the case of many samples of collodion cotton different fibres showed different degrees of nitration. Nitrated ramie fibres are more easily examined microscopically than cotton fibres, owing to the absence of torsion; the same optical relationships hold good. On denitration of highly nitrated ramie fibres by ammonium sulphide the changes in sign of the double refraction are gradually reversed until the optical properties of the original cellulose are restored. The pure ramie fibre has an index-ellipsoid similar to that of an optically bi-axial positive crystal, whilst that of the highly nitrated fibre corresponds to a similar negative crystal. Stretching of the fibre causes in both cases an accidental positive double refraction. J. F. B.

Rapid Testing of Potato Starch for Finishing Purposes. V. Ravizza. (*Färber-Zeit.*, 1913, **24**, 416-419; through *Chem. Zentralbl.*, 1913, II., 2170.)—For the testing of potato starch for stiff finishes and sizing twine, the viscosity of the paste is measured. A viscosimeter is employed, consisting of a 50-c.c. pipette, with the bulb surrounded by a steam jacket, so that the temperature of the paste is maintained between 96° and 98° C.; 5 grms. of potato starch are stirred with 25 c.c. of warm water, poured into 200 c.c. of boiling water, and washed in with 25 c.c. of water; the paste is stirred and used at once for the estimation of viscosity. The viscosity of the paste reaches its maximum after twenty to thirty minutes on the water-bath (sometimes after fifteen, sometimes only after forty minutes); it then falls somewhat more slowly than it rose, until after about one hour, when the changes become very small; no change is observed after one and a half hours. The viscosity at the end of the heating was practically the same for all the samples studied, but the highest *maxima* were shown by those starches which give the best results for stiff finishes. The curve of the changes in viscosity is characteristic for each particular sample. The value of the highest viscosity of the paste stands in geometrical ratio to the concentration and cannot be measured with concentrations over 2·5 to 3 per cent. The increase in viscosity is due to the further swelling of the starch granules, whilst its fall is due to the bursting of the membranes of amylopectin and the solution of the amylocellulose. Organic acids, to the amount of 2 to 5 c.c. of $\frac{N}{10}$ solution in a paste of 5 grms. of starch in 250 grms. of water, are sufficient to hinder the formation of the paste; acetic acid has the weakest action, and citric acid the strongest. Pectic acid derived from grey flax yarns has a strong liquefying influence which accounts for the practical observation that linen yarns break down the sizing baths.

J. F. B.

Estimation of Urea. A. Jolles. (*Biochem. Zeitsch.*, 1913, **57**, 414-420.)—The volume of nitrogen evolved on decomposing urea solutions with hypobromite can be made sufficiently quantitative for analytical purposes by employing either a hypobromite solution containing an excess of alkali (150 grms. sodium hydroxide, 25 grms. bromine, 250 c.c. water), and an approximately 2 per cent. urea solution, or by employing a less alkaline hypobromite solution (100 grms. sodium hydroxide, 25 grms. bromine, 250 c.c. water), and adding potassium ferricyanide, in which case the concentration of the urea may be between 2 and 3 per cent. The latter method is preferable because it gives better results and does not necessitate using urea solutions of such definite concentrations.

E. W.

Titration of Potassium Xanthate. B. Holmberg. (*Ber.*, 1913, **42**, 3853.)—The xanthate is decomposed by acid, forming alcohol, carbon disulphide, and potassium salt, and the excess of acid titrated back, phenolphthaleïn or methyl orange being used as indicator. About 0·2 grm. of potassium xanthate was weighed, dissolved in 20 c.c. water, and excess of $\frac{N}{10}$ hydrochloric acid added. The decomposition of the xanthate was complete in a few minutes; the indicator was then added and the titration carried out with $\frac{N}{10}$ barium hydroxide. One equivalent of acid is neutralised by one of xanthate. Mercury may be estimated by this method, provided that it is present in the mercuric condition, and that no other metals precipitated by potassium xanthate, or oxidising agents such as nitric acid, are present. A weighed quantity of mercuric chloride in neutral solution was treated with excess of potassium xanthate of known amount; the insoluble mercuric xanthate separating was not removed by filtration, the excess of potassium xanthate being determined in the liquid as above. The results appended showed satisfactory agreement with theory. O. E. M.

INORGANIC ANALYSIS.

Michrochemical Detection of Aluminium. F. Rathgen. (*Zeitsch. anal. Chem.*, 1914, **53**, 33-37.)—When any material containing aluminium is treated with ammonium fluoride and sulphuric acid, and the mixture is heated to expel excess of acid and finally ignited, hexagonal crystals of corundum are formed. These crystals are sometimes 0·1 mm. in diameter, but more often a magnification of 300-400 is necessary before they can be recognised with certainty. As only iron and chromium give rise to similar crystals, which may be distinguished by their high colour, it is suggested that the following affords a good test for aluminium. The material is treated with 0·1-0·2 grm. ammonium fluoride and 4-5 drops sulphuric acid, and, after expulsion of the acid, the residue is raised momentarily to a red heat. When cold, it is transferred to a drop of water on a slide and examined microscopically.

G. C. J.

Method of Estimating Very Small Quantities of Boric Acid in Organic Substances. G. Bertrand and H. Agulhon. (*Comptes rend.*, 1913, **157**, 1433-1436.)—The method depends on the coloration of strips of turmeric paper immersed partially in the boric acid solution. From 10 to 20 grms. of animal substance, or about 1 grm., of vegetable substance, are mixed with a small quantity of pure sodium carbonate, and incinerated; the ash, which need not be white, is treated with from 5 to 10 c.c. of phosphoric acid solution, transferred to a flask, 20 c.c. of methyl alcohol are added, and the mixture is distilled, the distillate being collected in a platinum basin containing 5 drops of $\frac{N}{1}$ sodium hydroxide solution. A further quantity of 10 c.c. of methyl alcohol is then added to the contents of the flask, and the distillation is continued. The distillate is now evaporated to dryness, the residue is dissolved in 4 drops of $\frac{N}{10}$ hydrochloric acid and 0·5 c.c. of water, and the solution is rinsed into a small test-tube with a small quantity of water, so that the solution measures 1·5 c.c. A strip of turmeric paper, 45 mm. long and 3 mm. in width, is placed down the side of the tube, so that the lower end dips into the solution, and similar strips are placed in a series of test-tubes containing known quantities of boric acid dissolved in hydrochloric acid; the test-tubes should be of such a size that about 15 mm. of the paper strip extends beyond the mouth. The tubes are then placed in an oven heated to 35° C., and after three hours the strips are observed. A red or orange coloured zone appears at the upper end of the strip, the length of the zone depending on the quantity of boric acid present. For instance, 0·1 mgrm. of boric acid yields a zone 8 mm. in length, whilst 0·001 mgrm. gives a zone of about 2 mm. If, instead of heating to 35° C., the test-tubes are maintained at the ordinary temperature, the colorations do not reach their full intensity until after the lapse of about twenty-four hours. W. P. S.

New Reaction for Boric Acid or Methyl Alcohol. E. Pieszczek. (*Pharm. Zeit.*, 1913, **58**, 850-851; through *Chem. Zentralbl.*, 1913, II., 1941.)—Methyl alcohol, free from formic acid, gives the green flame coloration with boric acid compounds without the addition of sulphuric acid. The coloration is obtained by mixing finely powdered borax with methyl alcohol in a dish, allowing one or two minutes for combination to take place, then igniting the spirit. The reaction may serve for distinguishing between methyl and ethyl alcohols, and the presence of 5 to 10 per cent. of methyl alcohol in mixtures may be detected, especially if the observation be made in the dark. J. F. B.

Estimation of Chromium as Chromic Oxide. G. Rothaug. (*Zeitsch. anorg. Chem.*, 1913, **84**, 165-189.)—Souchay in 1865 and Wilm in 1879 reported that chromium was invariably over-estimated when precipitated as chromic hydroxide and weighed as chromic oxide. At a later date Treadwell (1882) and Stock and Massaciu (1901) claimed that the method was exact. Though the large errors of the earlier workers were possibly due to the less-resistant glass available in their day, it is now shown that the method as ordinarily carried out is not exact, and that this is due to the formation of chromic acid. When the ignited precipitate is leached out with water, chromion and chromation pass into solution. The error in ordinary circumstances averages 0·75 per cent., but varies from 0·1 to 1·4 per cent. It is shown that oxidation is most rapid at about 300° C., more than ten times as rapid as at temperatures below 200° or above 400° C. By conducting the drying and ignition with due regard to the facts, the average error may be reduced to 0·2 per cent., but individual results may be 0·6 per cent. in error. Ignition in a Rose crucible in a current of hydrogen, allowing to cool in the gas, gives exact results. G. C. J.

Electro-Analytical Studies. B. P. Richardson. (*Zeitsch. anorg. Chem.*, 1913, **84**, 277-318.)—It is shown that arsenic must be in the arsenic condition if copper is to be separated electrolytically from arsenic. Suitable working conditions are described.

In order to obtain bismuth in satisfactorily adherent condition from a solution containing tartaric, citric, or lactic acid (to prevent formation of peroxide), it is necessary to reduce the effective voltage when the bulk of the bismuth is out. This must be raised again during the washing with dilute caustic alkali, which the author finds it essential to use if solution of bismuth during washing is to be wholly prevented.

Directions are also given for the separation of bismuth from arsenic, cadmium, and lead. The sharp separation from cadmium is discussed at length in view of the fact that this separation might be expected to present special difficulties.

The author advances the view that though the measurement of cathode potential has proved invaluable in working out new separations, once those separations have been shown to be possible and have been studied with the aid of C. P. measurements, it should be possible in many cases to dispense with the elaborate and costly appliances required for measuring cathode potential, and where possible this is certainly desirable. G. C. J.

Quantitative Mineralogical and Chemical Composition of the Granites and Gneisses. P. Tschirwinsky. (*Jahrb. f. Mineral.*, 1913, II., 238-242; through *Chem. Zentralbl.*, 1913, II., 1939-1940.)—A complete survey of the chemical relationships of all the known granites and gneisses and their mineral components is given. The analytical methods are critically discussed, and methods of calculating the mineral components from the analyses. For instance, the percentage of biotite in the granite is calculated by multiplying the sum of the $Fe_2O_3 + FeO(MnO) + MgO$ by the empirical factor 1·75. Methods of mechanical separation and the methods based on density are discussed. Of the petrographic methods, that of Delesse and Rosiwal is preferred in connection with a photographic microscope, observations being carried out on three sections at right angles to one another. A table of average analyses of a large number of minerals of this group is appended. The average percentage of quartz in granites is about 31. According to the author, pegmatites are produced in the granitic magma by a decrease of the quantity of quartz in the eutectic mixture and aplites by the increase of this component.

J. F. B.

Approximate Melting-Points of Some Commercial Copper Alloys. H. W. Gillett and A. B. Norton. (*U. S. Bureau of Mines, Technical Paper* 60, p. 12.)—Few figures on the melting-points of ternary and quaternary alloys have been published. References are given to such figures as are available and grounds advanced for regarding them as untrustworthy. The authors, who describe their experimental method in detail, have determined the melting-point of the following alloys with an error which probably in no case is as great as ± 10° C.

Alloy.	Percentage Composition.				Melting-Point, ° C.
	Cu.	Zn.	Sn.	Pb.	
Gun metal ...	88·0	2	10·0	—	995
Leaded gun metal	85·5	2	9·5	3	980
Red brass ...	85·0	5	5·0	5	970
Low grade red brass	82·0	10	3.0	5	980
Leaded bronze...	80·0	—	10·0	10	945
Bronze with zinc	85·0	5	10.0	—	980
Half yellow, half red	75·0	20	2·0	3	920
Cast yellow brass	67·0	31	—	2	895
Naval brass ...	61·5	37	1·5	—	855
Manganese bronze					870

These ten alloys represent a large proportion of the non-ferrous alloys in use in the ordinary foundry, and the above figures, in conjunction with well-attested figures for binary alloys, make it possible to fix the melting-point of many other alloys closely enough for most technical purposes. The following table for binary alloys is derived from curves due to Desch, Shepherd and Upton, and the melting-points are probably accurate to within ± 10° C.

Percentage Composition of Alloy.				Melting-Point, ° C.
Cu.	Sn.	Zn.	Pb.	
95	5		—	1050
90	10		—	1005
85	15		—	960
80	20		—	890
95		5	—	1070
90		10	—	1055
85	—	15	—	1025
80	—	20	—	1000
75		25	—	980
70		30	—	940
65	—	35	—	915
60	—	40	—	890
95	—	—	5	1065
90	—	—	10	1050
85	—	—	15	1035

G. C. J.

Iodimetric Estimation of Iodide Iodine. E. Müller and G. Wegelin.

(*Zeitsch. anal. Chem.*, 1914, **53**, 20-28.)—To avoid the troublesome distillation of iodine, Vincent (*J. Pharm.*, 1899 [vi.], **10**, 481) proposed to estimate iodide by adding excess of iodate and sulphuric acid, then adding excess of sodium bicarbonate, and finally titrating with a standard solution of thiosulphate. At a later date, Schirmer (*Arch. Pharm.*, 1912, **250**, 448) proposed to use borax instead of sodium bicarbonate to prevent interaction between the excess of iodate and the iodide formed in the final titration. Neither method yields accurate results. Both the bicarbonate and the boric acid solutions contain appreciable quantities of hypoiodite, and the thiosulphate is not oxidised quantitatively to tetrathionate, but some sulphate is also formed. This source of error can be avoided by substituting arsenious acid for thiosulphate, but the results are still inexact, due to adsorption of iodide by the iodine, which protects it from the action of the iodate.

Excellent results may be obtained by adding 10 c.c. of 10 per cent. sulphuric acid to the iodide solution contained in a stoppered bottle, then adding benzene and excess of iodate and shaking. The benzene dissolves the liberated iodine, and the iodide is quantitatively decomposed. After addition of 4 grms. powdered dry borax dissolved in water, the iodine is titrated with arsenious solution. The addition of iodide immediately before the titration is not essential, but increases the solubility of the iodine in the aqueous layer, and makes it possible to conduct the titration quickly without violent agitation. Bicarbonate can be used in place of borax without fear of loss of iodine with the carbon dioxide evolved, because this is washed by the benzene, but the frothing makes the method less convenient than the borax method.

G. C. J.

Study of Pemberton-Kilgore Method for Determination of Phosphoric Acid. P. L. Hibbard. (*J. Ind. and Eng. Chem.*, 1913, 5, 998-1009.)—The paper deals with the many sources of possible error in this method, which depends on precipitation of phosphoric acid as ammonium phosphomolybdate, solution of the well-washed precipitate in excess of standard caustic alkali, and titration of the excess of alkali, using phenolphthaleïn as indicator. In spite of the many sources of error, the author recommends the method to those who have many determinations to make, as it is more rapid than any other method, and gives results sufficiently accurate for many purposes if carried out by a chemist accustomed to use it and acquainted with the conditions to be avoided. For occasional work it is likely to prove slower, as well as much less accurate, than the gravimetric (molybdate-magnesia) method. High temperatures and prolonged heating of the precipitate tend to high results (precipitation of MoO_3). The official American method (fifteen minutes at 65° C.) gives as good results as any other combination. Higher temperatures for a shorter time, as recommended by some authors, may give satisfactory results; but there is greater risk of separation of MoO_3 unless the mixture be quickly cooled. Lower temperatures are safer, in that the time may be varied within fairly wide limits without effect on the result; but not only is the minimum time for completing a determination thus increased, but below 40° C. the precipitate separates in a form that filters badly. The influence of concentration of phosphoric acid, and the influence of nitric acid, ammonium nitrate, chlorides, sulphates, silica, iron, oxidising agents, organic matter, and excess of molybdate, are all dealt with fully, and the author's views supported by test numbers. When magnesium nitrate is used to destroy organic matter, the addition of some magnesia prevents loss of substance by deflagration, which is otherwise apt to occur. Silver phosphate is almost the only phosphate that can be prepared of definite composition, and its use is recommended for determining the titer of the solutions. It is difficult to prepare without some reduction of silver, but gravimetric determinations of silver and of phosphoric acid in samples prepared by the author showed that their composition corresponded to the formula Ag_3PO_4 far more closely than is required in a standard for the control of a volumetric method. Some fifty cognate papers are cited.

G. C. J.

Detection and Estimation of Very Small Quantities of Selenious Acid. J. Meyer and W. von Garn. (*Zeitsch. anal. Chem.*, 1914, 53, 29-33.)—The method depends on the liberation of iodine from hydriodic acid, and colorimetric comparison of the solution with a standard prepared from a known amount of selenious acid under approximately identical conditions. Two colorimeter cylinders are charged, one with 5 c.c. of the solution to be tested, the other with 5 c.c. of a standard solution of selenious acid. About 70 c.c. of water is added to each, together with a drop of gum arabic sol, which acts as a protective colloid in preventing the flocculation of the reduced selenium. An equal amount of acid, which must not be less than 5 c.c. of 5 per cent. hydrochloric acid, but may be any larger amount, is added to each cylinder, the contents of which are well mixed and made up to 99 c.c. Potassium iodide (1 c.c. of freshly prepared 50 per cent. solution) is finally added, and the mixtures are stirred and set in the dark for five minutes. They are then compared by altering the depth of one until, when viewed vertically, they appear identical. If the ratio of colour intensity exceeds 5 : 1, a fresh standard, more closely resembling the sample, is prepared. One part of selenious acid in 200,000 of solution can be estimated within 5 per cent., 1 part in 1,000,000 within 10 per cent., whilst 1 part in 10,000,000 or more can be detected. If the gum arabic be omitted, the results are irregular; holding up the reduced selenium not only avoids irregularities, but intensifies the colour, and so increases the sensitiveness as well as the accuracy of the method. G. C. J.

Rapid Estimation of Different Sulphur Compounds in Urine. R. Gauvin and V. Skarzynski. (*Bull. Soc. Chim.*, 1913, **13**, 1121-1127.)—The following modification of Raschig's benzidine method (*cf.* ANALYST, 1910, **35**, 331, 418) gives rapid and accurate results in the estimation of sulphuric acid. The standard solution is prepared by grinding in a mortar 40 grms. of pure benzidine with 50 c.c. of hydrochloric acid of sp. gr. 1·17, and making up the solution to a litre. For use this solution is diluted to 1 : 20. For the estimation of sulphates in, *e.g.*, 20 c.c. of urine (which contains about 0·04 to 0·05 grm.) the liquid is acidified with 2 c.c. of dilute hydrochloric acid (1 : 10) to prevent the precipitation of phosphates, oxalates, etc., and then treated with 350 c.c. of the dilute benzidine hydrochloride solution. The mixture is vigorously stirred, allowed to stand for fifteen to twenty minutes, and the benzidine sulphate filtered off, washed with 4 to 5 c.c. of water, stirred up with about 30 c.c. of water, and titrated with $\frac{N}{10}$ sodium hydroxide solution, with phenolphthaleïn as indicator. The total sulphur is estimated by boiling for twenty minutes a mixture of 20 c.c. of the urine with 0·2 grm. of potassium chlorate, 2 c.c. of hydrochloric acid, and 20 c.c. of water, then adding 1 c.c. of a 10 per cent. solution of sugar and continuing the boiling for some minutes. The liquid is next neutralised with dilute sodium hydroxide solution (turmeric as indicator), treated with 2 c.c. of hydrochloric acid and 350 c.c. of dilute benzidine solution, and the sulphuric acid estimated in the same way as before. The total oxidised sulphur (sulphates and sulphonated esters) is estimated by boiling 20 c.c. of the urine with 2 c.c. of strong hydrochloric acid and 50 c.c. of water for fifteen minutes, then neutralising the liquid, adding 2 c.c. of hydrochloric acid (1 : 10) and 350 c.c. of the dilute benzidine solution, and again estimating the sulphuric acid. The difference between the first and third estimations gives the amount of sulphur in the form of sulphonated esters, whilst the difference between the second and third estimations gives the unoxidised sulphur. The three estimations may be made in less than an hour, and the results are shown to agree closely with those given by precipitation with barium chloride. C. A. M.

Estimation of Thiosulphate in Presence of Sulphite. J. Bodnár. (*Zeitsch. anal. Chem.*, 1914, **53**, 37-41.)—When excess of silver nitrate is added to a neutral solution of a thiosulphate, silver thiosulphate is formed, but reacts rapidly with water with the production of silver sulphide and sulphuric acid. The reaction is said to proceed quantitatively, one molecule of sodium thiosulphate yielding one molecule of sulphuric acid. As silver sulphite is stable in the cold, it is proposed to estimate thiosulphate in admixture with sulphite by determining the acid set free on treatment with silver nitrate.

The neutral solution to be examined is poured slowly, with constant shaking, into excess of silver nitrate solution contained in a 100 c.c. flask. The contents of the flask are well shaken, allowed to stand two hours, made up to the mark, and filtered through asbestos. In 50 c.c. of the filtrate the excess of silver is precipitated by addition of sodium chloride, and, without filtering, the acid is titrated with caustic alkali, using methyl orange as indicator. Small quantities of thiosulphate can be estimated in presence of much sulphite with an error not exceeding 7 per cent. The error is apparently independent of the presence of sulphite, as a series of results with thiosulphate alone show errors ranging from 1 to 6 per cent. G. C. J.

Note by Abstractor.—The author recommends the above method for establishing the titer of thiosulphate solutions, but the test numbers show that the results are only roughly approximate.

Volumetric Estimation of Titanium. E. Knecht. (*Zeitsch. angew. Chem.*, 1913, **26**, 734-735.)—Neumann and Murphy have stated recently that the estimation of titanium by titration with methylene blue solution is the only method by which titanium can be estimated directly in the presence of silica, iron, aluminium, etc. The author does not agree entirely with this statement, for, although he has found the method to be trustworthy, titration with ferric solutions also yields accurate results. The latter method depends on the reaction $FeCl_3 + TiCl_3 = FeCl_2 + TiCl_4$, and is carried out as follows: A weighed quantity of from 0·5 to 1 grm. of the powdered mineral (rutile, etc.) is fused for ten minutes with 10 times its weight of alkali hydroxide; the fused mass is taken up with water, transferred to a beaker, and treated with a large excess of hydrochloric acid. The solution is diluted to 250 c.c., and 25 c.c. of it are placed in a flask which is closed with a stopper carrying a Bunsen valve. Hydrochloric acid and granulated zinc are then added, and when the iron salts, etc., are reduced completely and the zinc has dissolved, a zinc rod is placed in the solution and the latter is cooled. The rod is then rinsed with water, carbon dioxide is passed into the flask, and the solution is titrated with a standardised ferric solution, using potassium thiocyanate as indicator. Instead of titrating the cold solution in this way, it may be titrated while hot with methylene blue solution (*cf.* ANALYST, 1911, **36**, 98). A drop of methylene blue solution may also be employed as the indicator in place of thiocyanate in the titration with the ferric solution. Tungsten, molybdenum, vanadium, tin, and chromium must not be present. W. P. S.

Analyses of Waters from Thermal Springs in Palestine. A. Friedmann. (*Chem. Zeit.*, 1913, **37**, 1493-1494.)—The thermal springs in Palestine are found chiefly in the volcanic districts about the Jordan; those occurring at Tiberias, Lake of Galilee, yield water having a temperature of about 60° C., and containing from 28 to 32 grms. of total solids per litre, consisting mainly of sodium, calcium, and magnesium chlorides. Hydrogen sulphide is also present. The springs of El Hammi yield waters at a temperature of from 34° to 48° C., and containing from 0·71 to 1·21 grms. of total solids per litre; hydrogen sulphide is also present in these waters. All the waters are more or less radio-active. W. P. S.

APPARATUS, ETC.

Automatic Gas-Sampling Apparatus, with Some Observations on Sampling. T. Gray. (*J. Soc. Chem. Ind.*, 1913, **32**, 1092.)—Investigations on solutions of glycerol and of magnesium chloride as confining liquids showed that ordinary gaseous fuels and their products of combustion are sufficiently insoluble in them to be not seriously affected by contact with them for short periods, if they are sufficiently concentrated, and if agitation be avoided. To secure an average sample of gas, taken at a uniform rate over any specified period, an apparatus was constructed in which mercury is the confining liquid. The gas is drawn in a rapid current by a jet-pump through a tube communicating by a branch with an ordinary gas-sampling tube, to the lower end of which is attached a rubber tube and small levelling vessel. The mercury filling the sampling-tube is run out, drawing in a sample of the gas after it on lowering the levelling vessel, which is provided with a lateral overflow tube. The uniform lowering of the levelling vessel is accomplished by suspending it from a cord passing over pulleys to a float in a vessel into which water is run through a jet from a constant-level vessel; the rate is adjusted by altering the jet or the head of water. Alternatively, the mercury-levelling vessel may be substituted for the weight in a weight-driven clock movement, the rate being adjusted by shifting the pendulum-bob, by gearing, or by using several sampling-tubes. O. E. M.

Zeiss Interferometer for the Analysis of Gases. R. Lucion. (*Bull. Soc. Chim. Belg.*, 1913, **27**, 343-346.)—The author has found Zeiss's interferometer to be a useful and accurate piece of apparatus for the estimation of any particular gas in admixture with one or more gases—for instance, air in commercial hydrogen, carbon dioxide in air, etc. The apparatus consists of two parallel tubes, 1 metre in length and 1 sq. cm. in section, illuminated at one end by means of an electric lamp and fitted at the other end with an eyepiece, divided scale, and a prism. When the two tubes contain the same gas the two fields observed are identical in tint, but if another gas is introduced into one of the tubes, the scale has to be rotated in order to make the tints match. The apparatus must be standardised with mixtures of gases of known composition; pressure affects the results obtained, and means are provided for obtaining the gas in the two tubes at equal (or atmospheric) pressure. A difference of pressure of 1 or 2 cm., of water does not, however, affect the readings to any appreciable extent, and the same may be said of differences of temperature between 18° and 21° C. Since water vapour behaves as a gas in the apparatus, the gases under examination should be dried before being introduced into the apparatus. The limit of accuracy of the apparatus in the estimation of carbon dioxide or methane in air is from ± 0·01 to 0·02 per cent. W. P. S.

Improved Thermo-regulator. W. M. Clark. (*J. Amer. Chem. Soc.*, 1913, **35**, 1888-1889.)—Thermo-regulators which depend upon an electric contact between mercury and a platinum wire are adversely affected by the adherence of mercury to clean platinum, thereby causing the breaking of the circuit to take place at an appreciably lower temperature than the "make"; this may be largely obviated by the substitution of nickel or "nicrome" for platinum. The preservation of a clean mercury surface is more difficult to manage, and the only certain way to do this is entirely to prevent the access of oxygen. The author describes a method of sealing off the regulator in a current of pure dry hydrogen, after first carefully boiling the mercury to expel all air and moisture. The nickel contact wire is sand-papered to a fine point, and by proper adjustment may be kept centred on the mercury meniscus. Such an apparatus has been used continuously for three months without requiring attention, and has proved extremely sensitive. H. F. E. H.

Determination of Viscosity. M. P. Appleby. (*J. Chem. Soc.*, 1913, **103**, 21-67.)—The author points out that it is more convenient to state accurate relative results as such, and not as absolute values, since the determination of relative viscosity is free from the errors unavoidable in absolute determinations. The deviation from Poiseuille's law is due to formation of eddy currents at changes of bore, and not to the emergence of the liquid from the capillary with a definite velocity. For a variation of time of flow from 874·7 to 391·5 sec., it has been shown that the deviation of *pt* does not exceed 0·04 per cent. If Bingham's contentions (*J. Chem. Soc.*, 1913, **103**, 959) were valid, a deviation of 3 per cent. would have been obtained. The three difficulties in maintaining constant conditions, on which Bingham bases his objection to the slow-flow viscometer are cleanliness, constancy of filling, and temperature regulation. The vitiation of experiments by the intrusion of dust was due to the degree of accuracy sought by the author; the constancy of filling was such that the maximum variation in six fillings by a pipette was only 0·002 per cent.; the temperature regulation was effected by means of a Lowry spiral regulator actuating a post-office relay through which runs the current for the heating lamp, and could be reduced to a variation of 0·002° C. To prevent sparking at contacts a condenser of two aluminium plates in tap-water was placed across each gap. By attaching the regulator to the stand carrying the stirrer the surface of the mercury was kept just trembling, and sticking of mercury and platinum thus avoided. A slit was cut in the side of the cork carrying the upper contact of the spiral regulator, so that the pressure inside and outside was the same. O. E. M.

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REVIEWS.

THE PLANT ALKALOIDS. By THOMAS ANDERSON HENRY, D.Sc. (Lond.), Superintendent of Laboratories, Scientific and Technical Department, Imperial Institute, London. I. and A. Churchill. 1913. Pp. 466. Price 18s. net.

This work will prove of great interest and value to all interested in the plant alkaloids. The author has dealt with a difficult subject in as scientific a manner as is possible with the present state of our knowledge of this important class of compounds, many of which have been little investigated. He points out the difficulty of giving an exact definition of the term "alkaloid," and he uses the generally understood description—viz., "A relatively complex basic substance, occurring naturally, and possessing some physiological action." He also shows that this definition cannot be rigidly applied, and draws attention to the close association between vegetable and animal alkaloids.

The author attempts a classification of the plant alkaloids based on their chemical constitution. He divides them into eight groups, each group being characterised by the particular nucleus possessed by its respective members—*e.g.*, Group I. is the Pryolle group, Group II. that of Pyridine, Group IV. that of Quinoline, etc. The ninth group comprises alkaloids of unknown constitution, and the description of these occupies 111 pages—*i.e.*, about a quarter of the book. It is thus seen what a large amount of research remains to be done before the constitution of many important alkaloids has been determined.

The manner of dealing with the description of the alkaloids is excellent. In every case practically all the important references to original papers are given with the dates, so that the work has a considerable historical interest. The constitution of these alkaloids where known is clearly demonstrated by graphic formulæ, and the reactions by which the formula has been deduced in each case are pointed out. Of especial interest and importance are the references to the American, English, and German Pharmacopœias, the important tests and methods of estimation of which are given in full detail. The physiological action of the various alkaloids is accurately and fully given. The author, in his preface, acknowledges the help he has received in this part of the work from Professor Cushny's "Textbook of Pharmacology and Therapeutics."

The tests for the various alkaloids are given. In some cases these tests might be given in greater detail and more fully—for example, under Morphine there is no reference to Frohde's test, and the iodic acid test is very incompletely described. No mention is made of the distinctive tests between morphine and its derivatives, heroin and dionin. The recently described methods of extracting morphine by means of phenol and cresols are not mentioned. Under the tests for strychnine it would have been well to mention the physiological test of producing convulsions by injection of the solution into the abdominal cavity or dorsal lymph sac of a small frog. This test is so delicate as to detect $\frac{1}{3000}$ grain of the alkaloid, and is of great value in toxicological work. The author includes the alkaloids of curare under the quinoline group with strychnine. So little is known of the constitution of these alkaloids that they might well be placed in Group IX. amongst the alkaloids of unknown constitution.

Dr. Henry is to be warmly congratulated on the scientific and able manner in which he has dealt with an important and difficult branch of chemistry. His work will prove of great value to those interested in organic chemistry, and it will be of especial value and interest to pharmacists, pharmacologists, and those engaged in toxicological chemistry.

W. H. WILLCOX.

Organic Chemistry for Advanced Students. Vol. II. By Julius B. Cohen, Ph.D., B.Sc., F.R.S. London: Edward Arnold. Pp. vii + 427. Price 16s. net.

Professor Cohen published the first volume of this work about six years ago, and instead of writing a systematic textbook of organic chemistry, discussed several topics of special interest; the same course has been followed in the second volume. The author states in the preface that the volume published in 1907 embodied the substance of a course of lectures given at that time to senior students; the course has since been modified, and the volume now before us is the outcome. Organic chemistry is evidently presented to Leeds students in an attractive manner, and the reader can but admire the detailed attention devoted by Professor Cohen to the matter treated.

The work is divided into five chapters. The first of these deals with the *valency of carbon*, and affords most interesting reading. The question of carbon possessing a lower valency than four is discussed; needless to say, the work of Nef and the subject of "triphenylmethyl" receive considerable attention. After dealing with the "double bond" and "free valency," Professor Cohen proceeds with an examination of the nature of valency, the ideas of Werner, Thomson, Stark, Abegg, and Bodländer coming in for attention; this forms a very noteworthy feature in a work professing to deal with organic chemistry.

Chapter II., on the Nature of Organic Reactions, occupies about 160 pages, and deals with a variety of subjects—catalytic processes, condensation, and ring formation may perhaps be specially mentioned. Whilst this chapter is the most "organic" in the book, a good deal of general theory is introduced.

The Dynamics of Organic Reactions form the subject-matter of the third chapter, which has been written by Dr. H. M. Dawson; this and the succeeding chapter on Physical Properties and Structure, form a distinctive feature of the book. The line that at one time separated the physical chemist from the organic chemist has fortunately been broken down, and an increasing number of workers are interesting themselves with the physics of organic compounds.

Colour and Structure are dealt with separately in Chapter V., Fluorescence and the Photo-Chemistry of Organic Compounds following in natural sequence. Though less than sixty pages in length, this chapter gives an excellent idea of the lines of thought followed by different workers.

Professor Cohen's book is got up in good style and is very free from misprints; it concludes with indexes arranged according to subjects and authors. A useful feature is the list of references given at the end of each chapter; these refer to standard works dealing with the subjects which have been treated, the references to individual papers are to be found in the usual manner in footnotes. The work will well repay careful attention, and one may hope that when Professor Cohen next revises his lecture course, another volume may be added to the series.

J. T. Hewitt.

MARCH, 1914. Vol. XXXIX., No. 456.

THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

The annual general meeting was held on Wednesday evening, February 4, in the Chemical Society's Rooms, Burlington House. The President, Mr. L. Archbutt, F.I.C., occupied the chair.

The minutes of the previous annual meeting were read and confirmed.

Messrs. E. Richards Bolton and Cecil Revis were appointed scrutators of the ballot papers for election of officers and Council for 1914.

The Hon. Treasurer, Mr. E. Hinks, B.Sc., presented the accounts of the Society for the year 1913.

These were adopted, and votes of thanks passed to the Hon. Treasurer, Hon. Secretaries, and Auditors, and to Mr. L. M. Nash for his work in connection with the Dining Club.

On the motion of the President, a vote of thanks was passed to the President and Council of the Chemical Society for their kindness in allowing the Society the use of their rooms at Burlington House during the past year.

Mr. H. F. Knight and Dr. H. P. Stevens were appointed Auditors of the Society's accounts for 1914.

The President delivered his annual address.

Mr. Bevan moved a hearty vote of thanks to the President for his address and for his services in the chair, and that his permission be asked to print the address in the Analyst.

The motion was seconded by Mr. Raymond Ross, and was carried by acclamation.

The President, having received the scrutators' report, announced the election of Officers and Council for 1914 as follows:

President.—A. Chaston Chapman, F.I.C.

Past-Presidents serving on the Council (limited by the Society's Articles of Association to eight in number).—L. Archbutt, F.I.C.; Edward J. Bevan, F.I.C.; Bernard Dyer, D.Sc., F.I.C.; Thomas Fairley, F.I.C.; Otto Hehner, F.I.C.; R. R. Tatlock, F.I.C.; E. W. Voelcker, A.R.S.M., F.I.C.; J. Augustus Voelcker, M.A., B.Sc., Ph.D., F.I.C.

Vice-Presidents.—George Embrey, F.I.C.; J. T. Hewitt, M.A., D.Sc., Ph.D., F.R.S.; H. Droop Richmond, F.I.C.

Hon. Treasurer.—Edward Hinks, B.Sc., F.I.C.

Hon. Secretaries.—P. A. Ellis Richards, F.I.C.; Rudolf Lessing, Ph.D.

Other Members of Council.—E. M. Chaplin, Ph.D., F.I.C.; J. H. Coste, F.I.C.; G. H. Gemmell, F.I.C.; Charles A. Keane, D.Sc., Ph.D., F.I.C.; G. D. Lander, D.Sc., F.I.C.; Thomas Macara, F.I.C.; L. Myddelton Nash, F.I.C.; Cecil Revis, A.C.G.I.; Harry Silvester, B.Sc., F.I.C.; Arnold R. Tankard, F.I.C.; Thomas Tickle, B.Sc., F.I.C.; John White, F.I.C.

An ordinary meeting followed the annual meeting, the newly-elected President, Mr. A. Chaston Chapman, F.I.C., being in the chair.

The minutes of the previous ordinary meeting were read and confirmed.

Certificates of proposal for election to membership in favour of Messrs. A. de Waele and R. H. Ellis, A.I.C., were read for the second time; and certificates in favour of Messrs. Robert Bickerstaffe, "Chilterns," Wooburn Green, Bucks, Works Chemist to Messrs. Thomas and Green, Ltd., and Consulting Chemist to Messrs. Thomas Bros. and Co., Temple Mills, Marlow; Thomas Henry Byrom, F.I.C., 58, Hornsey Rise, N., late Chief Chemist to the Wigan Coal and Iron Company; Sydney George Clifford, 3, Norman Villas, East Dulwich, S.E., Analytical Chemist to Messrs. Waterlow and Sons, Ltd.; Donald Richard Frazer, A.I.C., Zeitzer Strasse, 9, IV., Leipzig; and John McLaren, 7, Fauconberg Road, Chiswick, W., Chief Chemist to the Chiswick Polish Company, were read for the first time.

Messrs. A. M. Bailey, A.I.C., A. L. Barton, T. S. Haines, A.I,C., and A. G. A. Miller, B.Sc., were elected members of the Society.

The following paper was read: "Iodimetry of Arsenic, Copper, and Iron," by G. D. Lander, D.Sc., F.I.C., and J. J. Geake.

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ANNUAL ADDRESS OF THE RETIRING PRESIDENT.

In addressing you for the second time, at the end of my term of office, I wish in the first place to thank you all for the very kind support you have accorded me. Deeply conscious of my shortcomings, I have felt that you accepted my services in the spirit in which they were given, and it has, therefore, been a real pleasure to me to preside over our meetings. To the Council and to all the Officers of the Society I tender my most sincere thanks.

Since our last annual meeting we have elected 21 new ordinary members and 1 new honorary member, Dr. Beilby, F.R.S., to whom all chemists are indebted for his valuable services when President of the Institute of Chemistry, and for the keen interest which he takes in all matters relating to our profession. In 1913 we lost 5 members by death and 3 by resignation or non-payment of subscriptions. Our roll, therefore, now includes 14 honorary and 412 ordinary members.

Our losses by death, though one less than I had to record at the date of our last annual meeting, were heavier in 1913 than in 1912, and have, regrettably, included some of our most distinguished members. The death of Mr. Claudet, which occurred very early in the year, was referred to in my last address. Since then we have lost John Heron, a chemist of great repute in the brewing industry; Matthew Algernon Adams, one of our Past-Presidents, who, apart from his varied scientific attainments, will always be remembered among analysts as the ingenious inventor of the standard method of estimating the fat in milk; James Carter Bell, who, like Dr. Adams, was one of the earliest Public Analysts appointed, and the contributor of many valuable papers to our journal; and Julius Lewkowitsch, the talented author of the standard treatise on oils, fats, and waxes, and one of our foremost technological chemists.

The death of Dr. Lewkowitsch, which I deeply regret as a personal friend, apart from the great loss to chemical science, reminds us again of the rapid advance which has been made in recent years, and is being made even more rapidly, in the chemistry and technology of oils, fats, and waxes. I think it has been fortunate for us that Lewkowitsch settled here and devoted himself to this branch of study, as we have had the advantage of the simultaneous publication in our own language of his "Technology," which has become so indispensable to all analysts. It will, I fear, be difficult to fill the gap caused by his death, and to find a successor so well qualified to carry on this important literary work.

During the past session, 34 papers have been read at our meetings, and 37 original papers altogether have been published in our journal, of which 13 are on food and drugs, and 14 on general analytical subjects. Although none of these papers marks any great advance in analytical methods, many of them will be found of great value to analysts, and we are much indebted to their authors for contributing them to our proceedings. I hope that it may some day be found possible to print advance proofs of the papers read at our meetings, and distribute them among members interested, as is done by the Engineering and Metallurgical Societies. I think we should get better discussions of some papers, and the interest and value of our proceedings would be enhanced in other ways.

The following four papers read during the session contain the results of work carried out under the Society's Analytical Investigation Scheme:

"Antipyrin in Toxicological Analysis." By G. D. Lander, D.Sc., F.I.C., and H. W. Winter.

"The Determination of Tannin in Tea." By Henry L. Smith, B.Sc., F.I.C.

"The Properties of some Chlorhydrocarbons and their Uses in Chemical Analysis." By L. Gowing-Scopes.

"The Composition of Palm-Kernel Oil." By G. D. Elsdon, B.Sc., A.I.C.

The paper by Mr. Gowing-Scopes embodies a very large number of useful and painstaking observations, which he deserves our special thanks for having undertaken. Mr. Chapman informs me that other investigations are in hand, the results of which will be communicated to the Society in due course.

I cannot too strongly impress upon members the fact that our Hon. Secretaries are not only ready at all times to receive suggestions in regard to subjects for investigation, but that such suggestions will be cordially welcomed. Most members in the course of their practice meet with analytical problems requiring solution, which they themselves have not the time to investigate. Descriptions of processes are read in the technical journals, which seem to solve difficulties, but need verification, and ideas for new methods occur to members themselves, which they have not the time to work out. In our Universities and colleges, and in private laboratories, there are senior students and others who have the time and are anxious to undertake such work, and members can be brought into touch with them through the medium of our Hon. Secretaries. This is the essence of the scheme, in connection with which we have, as you know, a fund for defraying the out-of-pocket expenses of investigators. But this fund, which has been capitalised, does not at present bring in sufficient income to defray the cost of the work which is being done, and we want to increase this work. Therefore I appeal to all members to send suggestions to the Hon. Secretaries in regard to new subjects for study, and to help us to increase the fund to a sum which will bring in a substantial income. There is no better way in which we can increase the usefulness and prestige of our Society and our journal.

Our journal has cost rather more than last year, but has fully maintained its reputation as the only journal in the English language exclusively devoted to analytical chemistry. I had occasion last year to refer to the reviews of books which appear in its pages, and which are among the best of their kind which appear in the English journals. The abstracts are also, as a rule, very well done, and I am sure you will agree that the journal continues to reflect the greatest credit upon our Editor, Mr. Julian L. Baker, and the Publication Committee.

No further meetings have been held of the Conference called by the Chemical Society in 1912 to consider the possibility of taking steps to avoid duplication of abstracts, and I presume that the preliminary inquiries set on foot have not yet shown a sufficient unanimity of opinion in favour of proceeding farther in the matter.

Our Hon. Treasurer's report is not so satisfactory as last year. Expenses connected with the anniversary dinner, increased cost of our journal, extra clerical work, and special expenditure which will not recur, are the principal reasons why our balance at the end of 1913 was less than the year before. The increasing cost of commodities, and the general tendency to greater extravagance in expenditure, affect societies as they do individuals, and have to be carefully watched by those with approximately fixed incomes. This is fully realised by our Hon. Treasurer, Mr. Hinks, to whom we are much indebted for his services during the year. The continued fall in the value of securities is very disquieting, and has considerably reduced our assets temporarily, but a recovery has commenced which I hope will continue.

The Council has been busily engaged during the year. The Public Health (Milk and Cream) Regulations, issued by the Local Government Board in August, 1912, raised two questions of importance. In the first place attention was again directed to the continually increasing work of the Public Analyst, for which there has been as yet no suggestion from official quarters of increased remuneration, and in the second place the wording of the regulations left indefinite many points on which it was, in the opinion of the Council, desirable that further information should be obtained.

In regard to the first question, we are indebted to Mr. Cribb and Mr. Hehner for having prepared the first draft of a statement compiled jointly by representatives of this Society and of the Institute of Chemistry, and adopted by the Councils of these bodies, on the conditions of appointments of Public Analysts in which the duties, responsibility, and the qualifications required of the Public Analyst to-day are compared with the conditions existing in 1875. The growth of knowledge relative to the chemistry of foods, increasing complexity in the practices of adulteration and in the methods needed for their detection, the use of chemicals as preservatives (almost unknown in 1875), the search for which alone now involves in some instances more labour than did formerly the whole analysis, and the stream of Acts of Parliament, Government Departmental Regulations and Recommendations, with which the Public Analyst has to be familiar, and which are continually increasing his work—these and other matters are discussed in detail. It is shown that, notwithstanding the enormous increase in the work and responsibility of the Public Analyst, the remuneration which is considered by Local Authorities sufficient to pay for this work has tended to diminish rather than increase, and recommendations are made with a view to an amendment of this unsatisfactory state of affairs. A scale of minimum fees is suggested for the work done under the Sale of Food and Drugs Acts and the Fertilisers and Feeding Stuffs Acts, and it is urged that the position of the Public Analyst should be made secure so long as he continues to efficiently discharge his duties, and that he should be entitled to a suitable pension on his retirement. It is further pointed out that the relative official positions of the Public Analyst and the Medical Officer of Health should be kept distinct, and that the work which they severally perform for the benefit of the community, and in some of which it is desirable for them to confer with one another, will be much more efficiently and satisfactorily performed if the two officers are maintained on a footing of equality and equal responsibility to the Committee or Council under whom they serve. A copy of this statement during its preparation was sent to every Public Analyst for his criticism and remarks, and in its final form it has been printed in the Proceedings of the Institute of Chemistry, Part IV., 1913. Copies will be sent to the various Local Authorities concerned with the administration of the Sale of Food and Drugs Acts with a covering letter signed by the Presidents of the Institute and this Society. It is also proposed to send copies to the Local Government Board, and it is hoped that the latter will be willing to receive a deputation to further urge the views of the Institute and our Society. It will be in the recollection of members that the Local Government Board of England promised in 1908 that the subject should receive further consideration in connection with any fresh legislation, and the new Bill introduced by Mr. John Burns last August has given the Board the opportunity of redeeming its promise.

At the desire of the Council, our Hon. Secretaries addressed a letter to the Local Government Board last March, asking for information in regard to several points in the Board's Milk and Cream Regulations as to which it was thought that difficulty and misunderstanding were likely to arise, and in consequence of the legal decision in the Kensington appeal case a further letter was addressed to the Board in May. To these letters no formal reply has been received. But a supplementary circular was issued by the Board in October last (Milk and Cream, No. 2) which clears up most of the points which were in doubt, and it has been considered by the Council unnecessary to take any further steps at present. It is not quite clear whether the Regulations are intended to permit the addition of cane and beet sugar to cream, and even to ordinary milk, but this is a point which can be further dealt with when the necessity arises.

Mr. John Burns's new Bill, proposing to confer upon the Local Government Board the power, "after such inquiry as they think necessary," to "make regulations defining an article of food in any matter affecting its nature, substance, or quality," appears to be a tardy attempt to give effect in a limited way to the recommendation of the Select Committee of the House of Commons on Food Products Adulteration, appointed in 1894, that a Court of Reference should be established to, amongst other things, "prescribe standards and limits of the quality and purity of food." The need for such a Court was strongly advocated by Mr. Hehner in his evidence before the Committee, and has been urged by this Society for many years; but the Court should be a thoroughly representative body, and not merely the Local Government Board. The proposal of the Select Committee was that the Court should include, amongst others, the principal officer of the Government Laboratory, nominees of the Local Government Board and the Board of Agriculture, one or more analysts of repute, and representatives of the General Medical Council, the Institute of Chemistry, and the Pharmaceutical Society. It has never been suggested that the Local Government Board alone should constitute the Court, nor would such a proposal commend itself to any Public Analyst. I should like to read to you some remarks made by the late Sir Thomas Stevenson in his Presidential Address to this Society in 1897, the year following the publication of the Report of the Select Committee. In regard to the constitution of the Court of Reference recommended by the Committee, Sir Thomas said: "But as to the Court of Reference, the Committee do not give us in their recommendations all that we asked for, or all that justice demands. We are not to be directly represented in that body. Who are so familiar with foods, their composition and analysis, as the members of our own body, which embraces practising analysts of the highest distinction in every department of food and drug analysis? What body of men in this empire has more advanced scientific analysis than our Society? What British journal more teems with original analytical research than ours? The Chemical Society has practically long ceased to take interest in analytical work, except as to the means of determining formulæ; the Pharmaceutical Society only advances analysis in a limited field; and the Institute of Chemistry is an examining and qualifying body only. The Society of Public Analysts has now for a quarter of a century been the pioneer of analytical progress in this country. The result of the Committee's deliberations is a compromise. An unworthy proposal to exclude Public Analysts altogether from the Court of Reference was, however, happily rectified. There is no doubt that Public Analysts will sit in the Court of Reference, which would be an incomplete and incompetent Court without them. The Institute of Chemistry and its Council contain so many able and prominent chemists who are also Public Analysts that there is little doubt that the apparent unfairness of the exclusion of Public Analysts from a seat on the proposed Board will be rectified, unless, by some strange fatality, the members of the Institute of Chemistry, which is to be represented, be also excluded from the provisions of the forthcoming Bill. But that contingency is not likely to occur. For my part, I think that it is immaterial how the members of the Court are selected, provided that competent persons be secured whose decisions will command respect. Did I think the 'analysts of repute,' among whom the representative of the Institute of Chemistry finds a place, would not include Public Analysts, I, for my part, would vigorously protest against the finding of the Committee; but I think we may congratulate ourselves on having gained, though indirectly, all that we demanded as to a Court of Reference."

We can infer from the above how vigorously Sir Thomas Stevenson, a man of remarkably sound judgment, would have protested against a proposal to make the Local Government Board the sole arbiter in such a difficult subject. There is, however, a saving clause in Mr. Burns's Bill, viz., "after such inquiry as they may think necessary"; and under this clause a competent Court of Reference could be established to advise the Board. But, as Mr. Hehner states in a letter I have received from him, "in a matter which is vital to every manufacturer and vendor of every article of food or drink, touches every consumer, and can only be dealt with piecemeal after lengthy investigation, calling for the co-operation of scientific and technical men, it is essential that the details of the machinery by which the regulations are to be made be clearly and definitely stated in the Bill."

Having dealt with some of the matters which concern mainly the Public Analyst, I should like to say a few words in regard to my own branch of our profession. A chemist to a railway has very wide duties to perform. Foremost amongst them is the testing of the various materials of construction and stores. To enable this to be efficiently done, the chemist must have a thorough knowledge of the properties of materials, and the ability to distinguish between those that are essential and those that are not essential for the purpose of specification. The quantities of materials used by the railway are so large that practically everything is purchased under contract, and the basis of the contract is either a specification or a standard pattern or sample. The chemist's first duties are, therefore, to draw up, or assist the engineer in drawing up, the specification, and to see that the standard pattern or sample is of suitable quality; his next duty is to test the goods as they are delivered by the contractor, and see that they comply with the specification or are equal in every essential respect to the sample or pattern. The specification should be as simple as possible, consistent with procuring the class of goods required. Over-elaborate specifications increase the cost of goods, as the contractor becomes suspicious lest there be some point which it will be costly for him to comply with. But I am bound to say that greater elaboration is necessary nowadays than was formerly the case.

The articles covered by the term "materials of construction and stores" are very numerous, and include various descriptions of iron, steel, and ferrous alloys, copper and other non-ferrous metals and alloys, used in the construction of the permanent way, locomotives, and rolling stock; creosote for the preservation of timber; coal, liquid and gaseous fuels; various kinds of lubricants and illuminants; paints and colours, turpentine, soap and alkalies, rubber goods, water for steam-raising and for drinking, articles of food and drink sold by the refreshment department, and many other articles of less importance.

Another very important part of the chemist's duties are advisory, and concern the regulations and conditions necessary for the safe conveyance over the railways of explosives, inflammable goods, dangerous, corrosive, and poisonous chemicals;

questions connected with the safe storage of goods in warehouses, and the loading and unloading on the company's premises of tank waggons containing articles such as vitriol, naphtha, and motor spirit—all of which matters concern the safety of the persons and property of the public and of the railway company and its employees. Questions are continually arising in connection with new kinds of traffic, such as their nature and value, the category into which they fall, whether they are provided for under the existing classification of goods, or need new and special provision to be made for them; questions also relating to the correct or incorrect description of goods handed to the railway for conveyance, and the justice or injustice of claims made for damage to goods during conveyance or storage. I used sometimes to be asked what a railway company could find for a chemist to do; the difficulty would be to answer in what direction of its numerous ramifications the railway company is unable to find work for the chemist. Even the Secretarial Department has consulted me in reference to the discovery of an indelible ink, and I am bound to add that up to the present I have been unable to find an ink which satisfies their requirements.

I have said enough to show that the railway chemist, if he is to do his duty to his employers, and if his services are to be adequately performed, must be a man of special training and experience, and he should be well paid. When I was first appointed to my present position, some thirty years ago, there were very few railway chemists; and not even we ourselves, much less those appointing us, had any adequate idea of the work there was for us to do. Our duties and their importance are gradually becoming better appreciated, and the conditions are improving. As in the case of the Public Analyst, I feel that inadequate remuneration, where it exists, arises, not from any desire on the part of employers to underpay their technical staffs, but from imperfect knowledge of our work and what it involves. It falls to each of us, individually as well as collectively, to do his utmost to remedy this lack of knowledge.

In my address last year I referred to several matters requiring investigation. I have time now to refer again to only one of these. I mentioned that the National Physical Laboratory had the subject of Viscometry under investigation. A report has now been issued. It embodies the results of experiments carried out for the International Commission for the Unification of Tests on Petroleum Products, and deals with the standard viscometers used in this country and on the Continent, designed respectively by Sir Boverton Redwood and Dr. C. Engler. A description of the essential features of each of these instruments is given, and the magnitude of the errors due to variations in the initial level of the liquid, errors of levelling and in the volume of the measuring flask, are computed. The results are then given of a series of experiments with oils ranging in viscosity from 0·0208 to 5·15, showing that by means of a formula the viscosities in absolute measure of oils included within this range can be calculated with remarkable accuracy from their times of efflux from the Redwood viscometer. Ubbelohde has calculated a similar formula for the Engler viscometer, and from the two formulæ the ratio of the readings of the two instruments has been calculated. This report is most valuable as far as it goes. One point is not referred to, and that is the influence of variations in the position of the thermometer bulb, which should be rigidly fixed for each instrument, there being a very important slope in the temperature of the oil between the outside and the centre of the tube in both instruments when determining viscosities above or below that of the atmosphere. Large numbers of Redwood viscometers are in use, and it is well known that their indications do not always agree; the formula worked out at the National Physical Laboratory is, therefore, only applicable to the particular instrument tested and those which happen to be of the same dimensions. If this report is to be of practical value, what is now wanted is for the National Physical Laboratory to state that they are prepared for a reasonable fee to standardise any instrument sent to them, and issue a certificate giving the formula by which the absolute viscosities of oils can be calculated from the times of efflux. If this be done, the commercial expression of viscosities in absolute measure will only be a matter of a short time, and an immense advantage to all users of lubricating oils will have been gained.

In concluding these remarks, I desire once more to thank our Hon. Secretaries, Messrs. Chapman and Richards, for the very friendly help they have at all times given me, without which I could not have carried out the duties which have devolved upon me during my term of office. I congratulate the Society that our senior Hon. Secretary has been elected to succeed me in the chair. There is no member of this Society who is more keenly interested in its welfare than Mr. Chapman, and none more worthy to hold the premier position. His past services as Hon. Secretary and in connection with the Analytical Investigation Scheme which he initiated amply entitle him to this distinction, and the high position which he holds as one of our leading consulting chemists, a Past-President of the Institute of Brewing, an examiner to the Institute of Chemistry, and an original investigator who has contributed a large number of most important papers to our proceedings and those of other societies, will enable him to fill the chair with honour and dignity to the Society. Being also a Public Analyst, Mr. Chapman will be able to represent both branches of our profession.

We may also congratulate ourselves that Dr. Lessing has consented to accept the vacant honorary secretaryship.

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IODIMETRY OF ARSENIC, COPPER, AND IRON.

By G. D. LANDER, D.Sc., F.I.C., and J. J. GEAKE.

(*Read at the Meeting, February* 4, 1914.)

One of us in conjunction with A. E. Walden, B.Sc., had occasion, in connection with an investigation on the influence of copper salts on the solubility of arsenious oxide, to make numerous estimations of arsenic in the presence of excess of cupric salts. We found that the addition to the mixed solution of an excess of Rochelle salt and sodium bicarbonate enabled the iodimetric estimation of arsenic to be carried out directly, the alkaline cupric tartrate compound being altogether without action either on potassium iodide or iodine. The method is, however, not new. S. Avery and H. T. Beans (*J. Amer. Chem. Soc.*, 1901, **23**, 485-486) applied the same method to the rapid analysis of Paris green, with excellent results. These authors quote several good results, and remark that cuprous compounds appear to interfere with the process.

We have confirmed the method by a large number of test cases, and have extended it in several directions.

Copper and Arsenic.—We tabulate a few results showing the degree of accuracy of the estimation of arsenite in the presence of copper salts. The arsenite solution used was the same throughout, and of such strength that 10 c.c. equalled 8·2 c.c. of $\frac{N}{10}$ iodine. The solution of copper sulphate was of 20 per cent. strength, and that of cupric chloride was equivalent. The end-point with starch is exceedingly sharp:

Copper Salt.	Arsenite.	Iodine required.	Theory.
(1) 10 c.c. sulphate ...	10 c.c.	8·25 c.c.	8·2 c.c.
(2) 10 ,, ,, ...	20 ,,	16·50 ,,	16·4
(3) 10 ,, ...	5 ,,	4·10	4·1
(4) 10 chloride ...	20 ,,	16·40	16·4
(5) 10 ,, ...	10 ,,	8·15	8·2
(6) 10 ,, ...	5 ,,	4·15	4·1
(7) 10 ,, ...	1 ,,	8·25	8·2 $\frac{N}{100}$
(8) 20 ,, ...	1 ,,	8·30	8·2 $\frac{N}{100}$

These results, well within the limits of experimental error, serve to further confirm Avery and Beans' method (*loc. cit.*). Experiments 7 and 8 also show that the method is suitable for the determination of traces of arsenic in the presence of relatively large quantities of cupric salt. It is desirable to add the Rochelle salt before the bicarbonate, otherwise there is a chance of some oxidation of arsenite, with corresponding reduction of cupric oxide.

Copper and Arsenate.—The determination of arsenate in the presence of copper after reduction to arsenite is likely to possess more practical applications than that of arsenite. In this connection we may draw attention to some of the interesting points regarding the reduction of copper salts by sulphurous acid.

When a solution of cupric sulphate in the presence or absence of dilute sulphuric acid is saturated with sulphur dioxide gas, or mixed with sulphurous acid solution, and made alkaline, a precipitate of cuprous oxide is formed. If the solution is boiled free of sulphurous acid, there is not a trace of cuprous salt recognisable in the solution. This is in conformity with the well-known fact of the non-existence, or at any rate extreme instability, of cuprous sulphate.

When sulphur dioxide acts on dilute cupric chloride solutions in the cold, there is no change of colour, suggesting the persistence of the cupric ion. On warming, the blue colour is more or less completely discharged; and on then cooling, cuprous chloride separates from the solution. Whilst, then, the iodine thiosulphate titre of cupric sulphate is unchanged after boiling with sulphurous acid, that of cupric chloride is diminished in proportion to the extent of reduction. In illustration of this the following result may be quoted: 20 c.c. of a cupric chloride solution required 10·2 c.c. $\frac{N}{10}$ thiosulphate; the same quantity after partial reduction required 4·2 c.c. As we show later, there is a definite reaction between cuprous halides and iodine in the presence of tartrate and bicarbonate.

It follows from these remarks that the rapid method may be applied to copper-arsenate mixtures, provided that sulphuric be the only acid present. This end may be easily achieved by evaporation—*e.g.*, of copper nitrate and arsenic acid with sulphuric acid. The following results prove the availability of the process for cupric sulphate-arsenate mixtures: 20 c.c. of arsenate solution, after reduction by sulphurous acid, required 16·8 and 16·6 c.c. $\frac{N}{10}$ iodine respectively. In the presence of copper sulphate (about equivalent in quantity to the arsenate), 20 c.c. arsenate required 16·5 $\frac{N}{10}$ iodine, whilst in the presence of cupric chloride 17·2 c.c. were required; but, as will be shown later, the excess of iodine used in this case was the result of oxidation of the cuprous halide in an alkaline tartrate medium.

Arsenite mixed with Salts of Iron and Other Metals.—We have shown that the method is available for mixtures of arsenite with ferric, but not with ferrous, chromium, nickel, cobalt, bismuth, and zinc salts. The addition of Rochelle salt, essential in the cases of cupric and ferric salts, which liberate iodine from potassium iodide, is not so for bismuth and zinc salts.

The orange-yellow and alkaline tartrate ferric solution is without action either on potassium iodide or iodine, so that arsenite may be determined by means of iodine, as in the case of copper. The best procedure is to add Rochelle salt and then excess of bicarbonate to the mixed solution.

Using 20 per cent. solutions of the ferric and zinc salts, the following results were obtained:

Salt.	Arsenite.	Iodine required.	Theory.
(1) 10 c.c. ferric chloride	2 c.c.	16·50 c.c. $\frac{N}{100}$	16·4 c.c. $\frac{N}{100}$
(2) 10 ,, ferric alum ...	20 ,,	16·20 ,, $\frac{N}{10}$	16·4 $\frac{N}{10}$
(3) 20 ,, ,, ...	10 ,,	8·15 ,,	8·2
(4) 10 ,, zinc sulphate	10 ,,	8·15 ,,	8·2
(5) 10 ,, ,, ...	10 ,,	8·05 ,,	8·2
(6) 5 ,, zinc sulphate 5 ,, ferric alum ...	20 ,,	16·25 ,,	16·4

In Experiment 5 no tartrate was used. Zinc tartrate is in any case not easily soluble in bicarbonate.

In our experiments with bismuth we used about 1 grm. of the carbonate or chloride dissolved in a slight excess of hydrochloric acid, and obtained the following results:

Bismuth Salt.	Arsenite.	Iodine required.	Theory.
(1) 1 grm. with tartrate	10 c c.	8·20 c.c.	8·2 c.c.
(2) 1	20 ,	16·40 ,,	16·4
(3) 1 ,, no tartrate	10 ,	7·70 ,,	8·2
(4) 1 ,, ,, ...	10	6·50 ,,	8·2
(5) 1 ,, ,, ...	10	7·64 ,,	8·2

In Experiment 5 the addition, after attaining the end-point, of Rochelle salt brought up the titre to 7·94 c.c. It is evidently preferable to add Rochelle salt, for apparently there is some chance of arsenite being precipitated with the bismuth carbonate.

Antimony and Cupric, Ferric and Bismuth Salts.—As would be expected, antimony mixtures can also be determined similarly to the arsenite. Ten c.c. of the tartar emetic solution used was equal to 9·85 c.c. $\frac{N}{10}$ iodine:

Salt.	Antimony.	Iodine required.	Theory.
(1) 2 grms. copper chloride	10 c.c.	9·85 c.c.	9·85 c.c.
(2) 2 ,, ,, ...	20 ,,	19·70 ,,	19·70 ,,
(3) 10 c.c. ferric alum (20 per cent.) ...	10 ,,	9·80 ,,	9·85 ,,
(4) 1 grm. bismuth chloride	10 ,,	9·85 ,,	9·85 ,,
(5) 1 ,, ,, (no tartrate)	10 ,,	9·45 ,,	9·85 ,,

In Experiment 5 the addition of Rochelle salt necessitated the use of a further 0·45 of iodine, bringing the result to 9·9 c.c.

Iodimetry of Cuprous Copper.—We find that the reaction

$$CuX_2 + 2KI = CuI + I + 2KX,$$

which occurs in neutral or acid solution, is quantitatively reversible, in that after addition of tartrate and bicarbonate the cuprous ion is oxidised to the cupric by iodine. The reaction is slow by reason of the slight solubility of cuprous iodide, and is therefore best accomplished by adding an excess of standard iodine and titrating back with thiosulphate. A large excess of bicarbonate must be avoided for the reasons given later.

The following experiment illustrates the process: Twenty c.c. of a copper sulphate solution, acidified with dilute hydrochloric acid and treated with potassium iodide, liberated iodine equivalent to 10·1 c.c. of $\frac{N}{10}$ thiosulphate. To the resulting liquid there were added Rochelle salt, a slight excess of solid sodium bicarbonate, and 15·1 c.c. $\frac{N}{10}$ iodine. The excess of iodine was equivalent to 5·0 c.c. $\frac{N}{10}$ thiosulphate, the iodine used in oxidation being thus equal to 10·1 c.c.—that is, to the amount liberated on reduction.

Taking advantage of these facts, it is possible to directly estimate cuprous and cupric copper, when present together, and some analyses of commercial cuprous chloride are quoted below. With regard to the oxidation of cuprous chloride, it must be pointed out that it is very necessary to have that salt in a finely-divided state. When this is so, the chloride is converted to finely-divided iodide by potassium iodide, but if larger crystals of chloride are present they are acted upon somewhat slowly. Without doubt, cuprous chloride would be oxidised as readily, or more so, than the iodide, but it appears always to be converted into the iodide by reason of the greater insolubility of the latter.

0·1 grm. cuprous chloride was dissolved in dilute hydrochloric acid, boiled water added, cooled, and an excess of potassium iodide added. 4·6 c.c. $\frac{N}{10}$ thiosulphate were needed. Rochelle salt and a slight excess of solid bicarbonate were added, and then 15·2 c.c. $\frac{N}{10}$ iodine. The excess required 5·95 $\frac{N}{10}$ thiosulphate, so that 9·25 c.c. $\frac{N}{10}$ iodine was equivalent to the total cuprous chloride, and less 4·6 due to original cupric chloride, this gives 4·65 due to original cuprous chloride. A second similar determination gave cupric chloride equal to 4 4, and cuprous chloride equal to 4·85.

Calculating to Cu_2Cl_2 and to basic cupric chloride, these results correspond to—

Cu_2Cl_2	45·8 per cent.	47·7 per cent.
Cu_2OCl_2	48·9 „	46·8 „

figures which are satisfactory, bearing in mind the lack of uniformity of cuprous chloride samples, and the extreme ease with which cuprous chloride oxidises in solution.

Iodimetry of Ferrous Iron.—The reversal in alkaline tartrate solution of the reaction $FeX_3 + 3KI \rightarrow FeI_2 + I + 3KX$, on lines parallel to those observed with copper, is possible, but the results are not so satisfactory from the quantitative point of view.

Nevertheless we believe the oxidation by iodine of ferrous iron to be about as complete as the liberation of iodine by ferric iron, when the latter is not facilitated by heating. On liberating iodine by means of ferric chloride without heating, and then adding Rochelle salts and bicarbonate, 1 or 2 drops of thiosulphate are, as a rule, sufficient to destroy the excess of iodine remaining.

We made a large number of tests on solutions of ferrous sulphate, using an excess of iodine, and titrating back. When a ferrous salt is in presence of alkaline tartrate, there is no doubt a shift of ferrous ions from their ordinary condition to association with the hydroxy groups of the organic acid, and we hoped that by adding the tartrate, then the iodine, and then the bicarbonate, we might catch the ion in *statu nascendi.*

We quote a few results in order to indicate the general character of the results, and to draw attention to certain pitfalls in the use of iodine in the presence of bicarbonate :

Ferrous Sulphate.	Iodine.	Thiosulphate.	Iodine used.	Permanganate.	Difference.
(1) 20 c.c.	40·20	20·70	19·50	19·55	-0·05
(2) 20 c.c.	40·30	20·50	19·80	19·55	+0·25
(3) 20 c.c.	40·20	21·15	19·05	19·55	-0·50
(4) 20 c.c.	40·20	19·90	20·30	19·55	+0·75
(5) 20 c.c.	30·35	11·45	18·90	19·30	-0·40
(6) 20 c.c.	25·15	6·40	18·75	19·30	-0·55
(7) 20 c.c.	40·30	21·70	18·60	19·30	-0·70

Standing for varied periods, and gentle warming at 40° C., do not succeed in securing greater uniformity in the results. In Experiments 1 to 4 the expression "excess of solid bicarbonate" was interpreted in a liberal sense. The use of an excess of iodine followed by back titration is not a usual analytical method, and it is therefore probably not well known that iodine is acted upon by bicarbonate, notwithstanding the fact that a drop of iodine causes a fairly permanent blue colour with a strong solution of bicarbonate and starch. Having regard to the undeniable existence in bicarbonate solutions of minimal amounts of hydroxide, arising from hydrolytic dissociation, or, in the equivalent language of the ionic theory, to the presence of hydroxylion, it is to be expected that action might occur.

The following data show the nature of the variation likely to arise from this cause :

Iodine.	Sodium Bicarbonate.	Thiosulphate.
(1) 10 c.c.	nil	9·6 c.c.
(2) 10 ,,	about 0·2 grm.	9·6 ,,
(3) 10 ,,	,, 1·5 ,,	9·2 ,,
*(4) 10 ,,	,, 1·5 ,,	9·2 ,,
(5) 10 ,,	"large excess"	9·0 ,,

The liability to error is not so great when iodine is added to the bicarbonate solution drop by drop, as in ordinary arsenic, antimony, and tin estimations—thus :

Thiosulphate.	Sodium Bicarbonate.	Iodine.
(1) 9·6 c.c.	nil	10·00 c.c.
(2) 9·6 ,,	about 2·0 grms.	10·05 ,,
(3) 9·6 ,,	"large excess"	10·15 ,,

Kahlbaum's bicarbonate was used in these experiments, and it contained less than $\frac{1}{100}$ mgrm. of carbonate per 10 grms. of substance.

In all our experiments we have therefore had to take care that the excess of

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* *After standing for fifteen minutes.*

bicarbonate did not greatly exceed 0·2 grm. In our earlier iron experiments it is sufficiently clear that a lucky compensation of errors may occasionally lead to a very excellent result.

DISCUSSION.

Mr. H. F. KNIGHT said that the rapidity of this method appeared to be its great advantage. For the determination, for instance, of arsenic in commercial copper there was, as far as he knew, no other method which took less than six or eight hours—usually more.

Mr. RICHMOND asked whether accurate results could be obtained by this method when the quantity of arsenic was very small in relation to the copper.

The PRESIDENT remarked that all these iodine reactions were more or less reversible. The well-known reaction $FeCl_3 + HI \rightleftharpoons FeCl_2 + HCl + I$ might be regarded as typical. This reaction, it was well known, took place quite readily in the cold, it being only necessary to prevent the reversal by employing a large excess of iodide, and taking care to have but little hydrochloric acid present.

Mr. GOWING SCOPES said that in the determination of arsenic in steel he had observed some absorption of iodine by the bicarbonate, and always made a blank determination whenever a new batch of bicarbonate was taken into use.

Dr. LANDER, in reply, said that the arsenic could be satisfactorily determined by this method in a solution containing 2 grms. of copper sulphate and 1 c.c. of decinormal arsenic solution—*i.e.*, about 1 part of arsenic to 500 of copper salt. Centinormal iodine solution could be used, about 10 c.c. being required for such a quantity of arsenic as that just mentioned. He agreed with the President that there was no necessity for heating in a closed bottle. In a similar way, heating in a closed bottle used to be recommended for the liberation of iodine from potassium iodide by means of bichromate, but in that case also it was quite unnecessary. The titration of iron by the liberation of iodine was perhaps not quite so smooth a process as in the case of copper, and probably the relative insolubility of the cuprous compounds, which the President had mentioned, had a good deal to do with this.

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ERRATA.

Page 83, line 8, *for* "10 per cent." *read* "20 per cent."
" 90, " 29, *for* "J. Bang" *read* "I. Bang."
" 90, " 37, *for* "Straszner" *read* "Strassner."
" 104, " 14, *for* "Appleby" *read* "Applebey."

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

Estimation of the Alkaloids: Application to Preparations of Nux Vomica. A. Azadian. (*Schweiz. Wochenschr. Chem. Pharm.*, 1913, **51**, 761; through *Chem. Zentralbl.*, 1914, I., 431.)—The alkalimetric estimation of the strychnine alkaloids gives inaccurate results, while precipitation with silicotungstic acid is satisfactory. Ten grms. of the powdered sample are extracted in a Seiler apparatus with 25 grms. chloroform, 50 grms. ether, and 5 grms. ammonia, with frequent agitation, for two to three hours. This is repeated, after removal of the solution, two or three times. The united solutions are concentrated on the water-bath, treated with 10 per cent. nitric acid, shaken with 10 c.c. of dilute nitric acid and 20 c.c. of water, and, after running off the acid liquid, washed with water until free from acid. The ether and chloroform are removed by evaporation on the water-bath; after cooling, 10 c.c. of 5 per cent. silicotungstic acid and 10 c.c. of 10 per cent. nitric acid are added; the liquid is heated to boiling, and allowed to stand twenty-four hours in the cold. The precipitate is collected on a 7 cm. filter, washed free from acid, dried and ashed. The weight of the residue (WO_3SiO_2) multiplied by 0·498 gives the weight of alkaloid. O. E. M.

Note on the Fatty Acids and Detection of Arachidic Acid. S. Fachini and G. Dorta. (*Chem. Zeit.*, 1914, **38**, 18.)—For the separation of liquid from solid fatty acids, Varrentrapp's method of treating the lead salts with ether is generally employed, but for qualitative or preparation purposes it is somewhat tedious. The following process is based on the observation that under certain conditions of concentration the potassium salts of the solid acids are almost insoluble in aqueous acetone, whilst those of the liquid fatty acids remain in solution. The following procedure is adopted in the examination of the fatty acids from olive oil: 10 grms. of the fatty acids are dissolved in 90 c.c. of pure acetone at boiling-point; the clear, boiling solution is treated with 10 c.c. of aqueous $\frac{N}{1}$ potassium hydroxide and allowed to cool. The first crystals appear at 35° to 32° C., and tufts of needles are formed on standing for some time at 15° C. The mass is collected on a dry filter, drained by the pump, and washed with small portions of pure acetone. The fatty acids are liberated, purified if necessary with light petroleum, and used for the determination of melting-point and molecular weight. The identification of arachidic acid is performed as usual by crystallisation from 90 per cent. alcohol. By the addition of the potassium hydroxide in quantity insufficient to combine with all the fatty acids—*i.e.*, by fractional neutralisation—the arachidic acid may be separated and identified by a single precipitation. The method is very convenient for the separation of large quantities of solid and liquid acids and for the isolation of individual solid acids by fractionation. J. F. B.

Commercial Analysis of Beeswax. G. Buchner and R. Deckert. (*Zeitsch. öffentl. Chem.*, 1913, **19**, 447; through *Chem. Zentralbl.*, 1914, I., 430. See also Fischer, *Zeitschr. öffentl. Chem.*, 1913, **19**, 354.)—The method of Leys (*J. Pharm. Chim.*, 1912, **5**, 577) for the estimation of alcohols and hydrocarbons in beeswax is accurate and rapid. The acetyl number of the alcohols of ordinary beeswax is nearly constant at 122, and consequently Leys' separation of the alcohols and hydrocarbons may be avoided if the acetyl number of the mixture is determined, and from this and the constant 122 the quantity of alcohols, and hence of the hydrocarbons, is calculated. East Indian beeswax, containing only ceryl alcohol, is most accurately determined by this method; ordinary beeswax contains chiefly myricyl alcohol, with a small quantity of ceryl alcohol and an alcohol of unknown constitution. O. E. M.

Occurrence of Boric Acid in Milk and in Eggs. G. Bertrand and H. Agulhon. (*Bull. Soc. Chim.*, 1913, **13**, 824-827.)—The following quantities of boron compounds, expressed as mgrms. of boric acid per litre, were found to be present in different milks, the samples having been obtained under conditions which precluded accidental contamination with the acid: Human milk, 0·45; ass's milk, 0·55; cow's milk, 1·1. Boron was also detected in various eggs, the amount about 0·14 mgrm. of boric acid per kilo of fresh white of egg obtained from hen, goose and turkey eggs. The yolks contained considerably less, only 0·008 mgrm. of boric acid being found in 1 kilo of yolk from hen eggs. W. P. S.

Estimation of Boric Acid in Foods. G. Bertrand and H. Agulhon. (*Comptes rend.*, 1914, **158**, 201-204.)—The following quantities of boric acid were found to be present in various fruits and vegetables, etc., the figures expressing mgrms. of boric acid per kilo of dry substance: Apricots, 112·6; dried apricots, 56·3; cherries, 112·6; figs, 56·3; strawberries, 112·6; peaches, 112·6; pears, 28·1 to 56·3; apples, 28·1 to 56·3; raisins, 56·3; carrots, 112·6; haricot beans, 56·3; onions, 112·6; peas, 56·3; potatoes, 11·2; tomatoes, 56·3; wheat, 5·6. The method employed for the estimation has been described recently by the authors (ANALYST, 1914, 96). W. P. S.

Report on Cocoa and Cocoa Products. W. L. Dubois. (*U.S. Dept. of Agric., Bureau of Chem.*, 1913, Bull. 162, pp. 130-135.)—*Casein.*—Baier and Neumann (*Zeitsch. Untersuch Nahr. Genussm.*, 1909, **18**, 13) have proposed to estimate the casein in milk chocolate by extracting it from the defatted material with sodium oxalate, and precipitating with uranium acetate in acetic acid solution. Five investigators have worked out this method, and find it gives results as follows:

	Sample 1.	Sample 2.
Casein actually present in chocolate	3·91	2 82
Casein found (average of all results)	3·71	3·21

The method actually employed is as follows: Ten grms. of the defatted material are rubbed up in a mortar with successive portions of 1 per cent. sodium oxalate solution until 200 c.c. have been used. The portions are transferred to a 250 c.c.

flask, and heated very slowly until the solution comes to the boil. The volume is made up with hot oxalate solution, shaken frequently, and allowed to stand until the following day. It is then cooled, made up, and filtered (or better, centrifuged). To 100 c.c. of the filtrate are added 5 c.c. of a 5 per cent. solution of uranium acetate, and then, drop by drop, 1·5 c.c. of 30 per cent. acetic acid. The precipitate is separated by three successive centrifugal washings, using 5 per cent. uranium acetate solution containing 3 c.c. of 30 per cent. acetic acid in 100 c.c.; and then the nitrogen determined in the precipitate and calculated to casein, using 6·38 as the factor.

Milk-Fat.—This is calculated from the Reichert-Meissl number of the ether extract, the value for milk-fat being taken as 24, and that for cacao butter as 0·5. The following formula is employed:

$$\text{Percentage butter-fat in chocolate} = \text{percentage total fat} \times \frac{C - 0{\cdot}5}{23{\cdot}5}, \text{ where}$$
$$C = \text{Reichert-Meissl number of extracted fat.}$$

In the presence of a large amount of butter-fat fairly accurate results may be obtained by merely deducting the cacao butter value (0·5) from the value found, and calculating the remainder into butter-fat; but where the milk-fat is present in very small amount a considerable error results, and the above formula should be used.

Methods for determining starch, fat, and sugar, are also discussed, but present no novel features.

H. F. E. H.

Gru-Gru Oil. A. W. Knapp. (*J. Soc. Chem. Ind.*, 1914, **33**, 9.)—Gru-gru is the Trinidad name for *Acrocomia sclerocarpa.* The tree belongs to the same tribe as the cocoanut palm. The oil obtained from the kernels by hot pressing gave the following figures:

	Oil.	Insoluble Fatty Acids.
Sp. gr. 99°/15·5° C.	0·861	0·838
Melting-point	26·0° C.	24·0° C.
Titre	—	23·05° C.
Iodine value (Wijs)	19·4	20·3
Refraction at 40° C.	36·95	19·7
Saponification value	243·5	—
Reichert-Meissl value	7·2	—
Polenske value	13·9	—
Shrewsbury and Knapp ...	163·0	—
Neutralisation value	—	262
Mean molecular weight ...	—	214
Free fatty acids (as oleic) ...	0·62 per cent.	—
Colour	Very pale yellow.	White.
Odour and taste...	Like cocoanut oil.	Like cocoanut fatty acids.

The opinion is expressed that this is the same oil as Mocaya oil of Paraguay and the oil from the maccasuba palm of Surinam (*cf.* De Negri and Fabris, *Chem. Rev.*, 1897, 82; also Sack, *Inspectie van den Landbouw in West Indie*, Bulletin 5, 1906).

G. C. J.

Detection of Hexamethylenetetramine in Wine or Milk. L. Rosenthaler. (*Pharm. Zentralhalle*, 1913, **54**, 1154; through *Chem. Zentralbl.*, 1914, I., 80.)—The liquid to be tested is acidulated with dilute hydrochloric acid, and treated with mercuric chloride solution. If hexamethylenetetramine is present, a characteristic crystalline precipitate is formed. The addition of 10 per cent. of alcohol promotes the precipitation. White wines require no preliminary treatment. Red wines are decolorised with lead acetate in excess, the lead is removed with sodium phosphate, and the test applied to the filtrate. Milk is made acid with hydrochloric acid, shaken vigorously with excess of solid ammonium sulphate, and filtered; if the filtrate is not clear, the fat is removed with light petroleum. The method is only applicable to free hexamethylenetetramine; it is sensitive to 1 part in 500,000. A list of other precipitating agents and their degrees of sensitiveness is given, with a description of the precipitates obtained. O. E. M.

Estimation of Hop Bitter Substances by Cold Extraction with Carbon Tetrachloride. R. Seibriger. (*Woch. Brauerei*, 1913, **30**, 610; through *Chem. Zentralbl.*, 1914, I., 302.)—Ten grms. of the minced hops are shaken for two and a half hours with 200 c.c. of carbon tetrachloride; the mixture is then filtered, and 100 c.c. of the filtrate are mixed with 50 c.c. of neutral alcohol, and titrated with $\frac{N}{10}$ potassium hydroxide solution. The percentage of bitter substances is then calculated as described by Lintner (*cf.* ANALYST, 1898, **23**, 331). The moisture is estimated by drying 3 or 4 grms. of the minced hops at 105° C. for three hours, and the amount of bitter substances found is expressed as a percentage of the dry hops. W. P. S.

Tobaccos from the East Africa Protectorate. (*Bull. Imp. Inst.*, 1913, **11**, 587-600.)—Experimental trials of American and Levantine tobaccos were carried out at Kabete, and samples received in England in December, 1912, were analysed, with the following results:

"VIRGINIAN" TOBACCOS.

	East African Tobaccos.		Nyassaland Tobaccos.		
	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.
Moisture	5·02	4·92	9·9	8·9	8·8
Total alkaloids (nicotine, etc.) ...	3·20	5·15	1·2	2·2	1·5
Total nitrogen	3·84	4·46	1·3	2·2	1·5
Ash	22·67	17·21	14·4	8·1	8·7
Composition of ash:					
CaO	33·20	33·56	17·4	36·3	26·8
MgO	7·11	9·95	5·7	9·3	8·8
K_2O	22·43	22·30	30·0	22·8	32·6
SO_3	4·17	5·01	1·9	3·7	2·2
Cl	0·66	0·90	0·4	2·2	0·3

The East African tobaccos cited above were packed too dry, and were grown in an unfavourable season. Unless more favourable climatic conditions cause considerable modification in these tobaccos, the above figures, taken together with the aroma, etc., suggest the unsuitability of the soil for this class of tobacco. Undesirable factors in the analyses are the high sulphate and chloride content of the ash and the low potash.

The following "Levantine" tobaccos have defects of the same type as the "Virginian" tobaccos, defects probably due to their having been cultivated in a soil too rich for these types, in an atmosphere containing an excess of moisture, and partly to the unfavourable season (1912).

"LEVANTINE" TOBACCOS.

	East African Samples. "Levantine" Types.		Commercial Samples of "Levantine" Tobaccos.		
	Per Cent.	Per Cent.	Macedonian.	Greek.	Samos.
Moisture	7·20	6·92	9·4 —10·9	8·9 —10·6	9·9
Total alkaloids (nicotine, etc.)	0·88	0·82	0·91 — 2·9	2·2 — 3·1	0·79— 1·6
Total nitrogen ...	3·72	3·71	2·0 — 2·5	3·1 — 4·4	1·6 — 2·2
Ash	15·83	13·71	9·3 —13·2	11·8 —15·3	10·6 —14·8
Composition of ash:					
CaO	31·42	24·28	39·12—58·8	30·7 —45·7	40·0 —44·2
MgO	10·35	8·81	5·34— 7·9	1·7 —10·9	4·5 —13·3
K_2O	22·24	25·97	6·87—12·67	7·4 —20·6	7·3 —13·2
SO_3	5·90	5·82	2·57— 4·25	2·5 — 4·5	4·1 — 9·0
Cl	1·27	0·97	traces to 1·38	0·29— 1·9	traces to 3·4

H. F. E. H.

Formation of Sediment in Bottled Mineral Waters. Estimation of Silica, Iron, and Aluminium, in Mineral Waters. L. Dede. (*Chem. Zeit.*, 1914, **38**, 53-55.)—The sediment frequently found in bottles of mineral waters consists chiefly of hydrated iron oxide, alumina, silica, and calcium carbonate; its formation does not appear to be due to the presence of considerable quantities of iron and calcium salts in the water, but rather to the amount of dissolved alumina and silica or suspended clay. It is probable that ferrous bicarbonate is first of all oxidised, and that the resulting hydrated iron oxide carries down the alumina and silica together with certain other constituents. The presence of alumina and silica appears to be essential, since highly ferruginous waters free from these constituents yield only traces of sediment.

W. P. S.

Estimation of Tannin in Cider. C. W. Spiers. (*J. Agri. Sci.*, 1914, **6**, 77-83.)—The method at present chiefly in use for the estimation of tannin in apple-juice and cider is a modification of Löwenthal's method, and consists in titrating the cider directly with potassium permanganate in presence of indigo as an indicator. Permanganate is standardised by means of "pure tannin," it being assumed that both this and the tannin present in the apple are digallic acid and have the composition $C_{14}H_{10}O_9$. The results obtained by this method are only comparative, and those given by gravimetric methods, such as the hide powder method, are useless, since it is practically impossible to obtain a residue of constant weight by evaporation of the cider. The method finally adopted was a modification of that of Körner and Nierenstein (ANALYST, 1911, **36**, 123), which consists in detannising the solution to be analysed by means of casein, the total solids being estimated before and after this treatment. In this case, however, the tannin removed was determined by the difference in the permanganate titrations, as in the method of Neubauer (*Zeitsch. anal. Chem.*, 1871, **9**, 1), the casein removing nothing but the tannin from the cider or the tannin solution analysed. The casein employed must be free from fat, and for the permanganate titration ammonium oxalate is employed for standardisation, 1 grm. of which is equivalent to 0·4648 grm. of tannin. This value was obtained as the result of the analysis of a large number of different samples of "pure tannin." It was found that the strychnine method of Trotman and Hackford (*J. Soc. Chem. Ind.*, 1905, **24**, 1097), although not accurate in the case of gallo-tannic acid, will precipitate quantitatively the tannin in cider after careful neutralisation. The author hopes shortly to prepare the tannin of apples in a pure state, so that it will be possible by means of it to standardise both the permanganate solution and the strychnine precipitate. Figures for the tannin content of a number of bottled ciders (1912) are given, determined by direct titration and by the casein method. The agreement on the whole is fairly close, the values running from 0·04 to 0·59 per cent.

H. F. E. H.

Method of expressing Results of Wine Analyses according to the Latest Chemico-Physical Principles. W. I. Baragiola and C. Godet. (*Zeitsch. anal. Chem.*, 1914, **53**, 100-114.)—An ordinary Tyrolese red wine has been taken as an example, fully analysed, and the manner of calculating the results so as to indicate the state of combination of acid and basic ions is explained. The physical estimations included in the analysis comprise determinations of sp. gr., alcohol and extract; the concentration of hydrogen ions is estimated by the determination of the reaction-velocity constant with diazo-ethyl acetate (Bredig); the electrical conductivity is determined during titration with $\frac{N}{4}$ barium hydroxide (Dutoit and Duboux), whence the proportions of potassium sulphate and total titratable acidity are calculated. Incidentally observations are made of the rotatory power of the sugar extract; the refractive indices of the wine, the alcoholic distillate, and the solution of the extract; the viscosity of the wine; the colour value and the turbidity. Chemical estimations in the extract include—invert sugar, glycerol; free and combined organic acids, mineral matters, viz., the ash *minus* the CO_2 and O ions, nitrogenous matters excluding ammonium salts, ammonium salts including volatile bases with sodium hydroxide; and the sum of these is balanced against the total extract found as such. The alcohol: glycerol ratio is calculated; cane-sugar, if present, is estimated; the SO_4 ion estimated by titration is balanced against the SO_4 determined in the ash. The free and combined organic acids are each estimated singly, and expressed as equivalents of $\frac{N}{1}$ solution, and their sum is compared with the sum of the titratable acids and the non-titratable acids as calculated from the alkalinity of the ash. The cathïons and anions of the ash are each estimated separately. Chemico-physical balances of the cathions and the organic and inorganic anions are then drawn up, for which purpose two totally distinct methods of calculation are available, one due to Quartaroli, and the other to Dutoit and Duboux. The formulæ are based on the molecular concentrations of the acids and their dissociation constants; in the case of dibasic acids provision is made for the calculation of the ternary equilibrium between free acid, acid salt, and neutral salt. The results of the two methods of calculation are in excellent concordance. The total titratable acidity and non-titratable organic acids thus calculated are then compared with the totals found experimentally.

J. F. B.

Malt Wine. P. Kulisch. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1913, **26**, 705-727.)—Malt wine is prepared by extracting crushed malt with hot water, whereby a more or less complete saccharification of the starch takes place, adding sugar to the extract, and fermenting the solution by the addition of brewery or distillery yeast. From 6 to 8 kilos of malt and from 8 to 15 kilos of sugar are used for the production of 100 litres of the wine. The fermented liquid is submitted to the usual filtering and clarifying operations, and is ready for consumption within about three months. Seventeen samples of malt wine gave the following results on analysis, the figures expressing grms. per 100 c.c.: Alcohol, 4·71 to 7·66; total solids, 1·46 to 4·65; ash, 0·056 to 0·19; total acidity (as tartaric acid), 0·27 to 0·61; volatile acidity (as acetic acid), 0·01 to 0·15; tartaric acid, none; citric acid, none; lactic acid (estimated in 5 samples only), 0·23 to 0·37; reducing sugars, 0·10 to 1·67; glycerol (11 samples), 0·3 to 0·5. The acidity develops spontaneously, and doubtless depends on the rate of fermentation.

W. P. S.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Modification of Teichmann's Test for Blood. C. T. Symons. (*Biochem. J.*, 1913, **7**, 596-599.)—The benzidine test for blood is not characteristic. The spectrum and corpuscle tests fail in many cases, as does the ordinary method of using Teichmann's test. The use of a mixture of sodium iodide and lactic acid is recommended for the latter in place of sodium chloride and acetic acid. A 1 per cent. solution of freshly prepared sodium iodide in lactic acid (sp. gr. 1·21) is used. This solution becomes brown on keeping, but the change does not appear to impair its action—at least, for some months. The fragment of stain is covered with the solution, the cover-glass placed in position, and the whole slowly warmed on an asbestos mat to such a temperature that the solution is just about to boil under the cover-glass. In most cases five minutes' treatment is sufficient to produce crystals, but if necessary the heating must be continued and more solution allowed to run under the cover-glass. The resulting crystals differ very much in size, being usually rather small, but dark and perfectly characteristic. E. W.

Estimation of Cholesterol and Oxycholesterol by the Method of Autenrieth and Funk. E. Schreiber. (*Münch. med. Wochenschr.*, 1913, **60**, 2001; through *Chem. Zentralbl.*, 1914, I., 78.)—A substance which gave the Liebermann colour reaction, but was not precipitated by digitonin, was discovered by Autenrieth and Funk in blood which had undergone three months' putrefaction. Oxycholesterol behaves in a similar manner. It may be estimated by the method of Autenrieth and Funk. In one portion the total cholesterol is determined colorimetrically; in the other the cholesterol is precipitated by digitonin. The filtrate is concentrated and extracted with ether; the extract is cooled with ice, and the separated excess of digitonin removed by filtration. The ether is driven off; the residue is dissolved in chloroform and again estimated colorimetrically. The quantities of the two substances are calculated from these two results. At least 10 c.c. of blood should be taken, as the quantity of oxycholesterol present is small. In any case the chloroform solution should be tested qualitatively for cholesterol by the acetic-sulphuric acid reaction or spectroscopically. O. E. M.

Fermentation Test at 46° C. as an Aid in Water Examination. C. Eijkman. (*Zentralbl. Bakter. u. Parasitenk.*, 1913, II., **39**, 75-80; through *Chem. Zentralbl.*, 1914, I., 183.)—A defence of cultural experiments at elevated temperatures as a test for the presence of organisms whose natural habitat is the intestinal tract of warm-blooded animals. The argument that there are genuine strains of *B. coli* that will not tolerate temperatures of 46° or even of 37° C. is met by the statement that the water examiner is less concerned with the identification of floral species than with determining the origin of the organisms present, and incubation at high temperatures does tend to encourage intestinal types and suppress organisms whose natural habitat is water. G. C. J.

Determination of Glycogen in Yeast. F. Schönfeld and E. Kunzel. (*Woch. Brauerei*, 1913, **31**, 9-12; through *J. Soc. Chem. Ind.*, 1914, **33**, 155.)—The method is a modification of that described by Pflüger (*Arch. ges. Physiol.*, 1909, **129**, 362) for the determination of glycogen in animal tissues. The yeast is washed with water for a short time and pressed. These operations should not be unduly prolonged, for the glycogen-content of the yeast is very liable to change. From 10 to 15 grms. of the yeast are heated with 25 c.c. of 60 per cent. potassium hydroxide solution for three hours in a bath of boiling water. The cooled product, washed into a larger vessel with 50 c.c. of water, is mixed with 200 c.c. of 96 per cent. alcohol, and left over night. The supernatant liquid is decanted, with as little solid matter as possible, through a filter connected with a pump. The residue, treated with a few drops of brine, is washed with 66 per cent. alcohol (100 to 150 c.c.) until the washings are colourless, then twice with absolute alcohol and twice with ether. The glycogen is redissolved by heating with water for some time, the solution is filtered into a

200 c.c. flask, neutralised with hydrochloric acid (sp. gr. 1·19), and made up to the mark. It is then heated in a bath of boiling water for three hours with 10 c.c. of hydrochloric acid (sp. gr. 1·19), cooled, neutralised with a 60 per cent. solution of potassium hydroxide, again made up to 200 c.c., and well mixed. The quantity of dextrose in 25 c.c. of the solution is determined by Allihn's method: 1 grm. of dextrose corresponds to 0·927 grm. of glycogen. A bibliography relating to yeast-glycogen is appended to the paper.

Detection of Decomposition in Meats. D. Ottolenghi. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1913, **26**, 728-758.)—The rate at which decomposition proceeds in meats may be ascertained by estimating the amino-acids formed, since the quantity of the latter increases in proportion to the decomposition. Sörensen's method (ANALYST, 1908, **33**, 19; 1910, **35**, 211) is employed for the estimation, but the titration is preferably carried out in four stages. The serum containing the amino-acids is obtained by heating 30 grms. of the finely divided meat in a closed tube for thirty minutes at 100° C.; the serum is treated with barium chloride and barium hydroxide, and filtered as described (*loc. cit.*). The filtered liquid is rendered neutral to litmus-paper, phenolphthaleïn is added, and then $\frac{N}{5}$ sodium hydroxide solution until a faint red coloration is obtained (Stage 1); the addition is continued until a deep red coloration is produced (Stage 2); neutral formaldehyde solution is now added and the titration continued to faint red (Stage 3), and deep red (Stage 4) colorations. The quantity of amino-acids (substances which may be titrated in the presence of formaldehyde) is thus obtained, and gives a measure of the degree of proteolysis which has taken place in the meat. Meat which may be considered to be fresh or well kept yields from 190 to 320 mgrms. of amino-acids per 100 grms. of dry substance, whilst meat which has commenced to decompose gives more than 350 mgrms. of amino-acids. The ratio of the amino-acids titrated in the first stage to the total quantity also affords some indication as to the freshness of the meat; in the case of fresh meat this ratio varies from 1 : 3·4 to 1 : 3·9, but becomes much smaller when decomposition sets in. A method proposed by von Fürth and Lenk (ANALYST, 1912, **38**, 455) was found by the author to be unsatisfactory, but a test described by Chodat (*Arch. Sci. Phys. Nat.*, 1912, **33**, 20), depending on the violet and red colorations obtained when amino-acids are treated with tyrosinase dissolved in *p*-cresol solution, yielded trustworthy results in the detection of these acids in decomposed meats.

W. P. S.

Valuation of Pepsin. O. Frey. (*Zeitsch. Allg. Österr. Apoth.-Ver.*, 1913, **51**, 593, 611, 623, 635, 649, 659; through *Chem. Zentralbl.*, 1914, I., 430.)—As the result of numerous experiments, the author sets up the following standard: In presence of 10 c.c. $\frac{N}{1}$ hydrochloric acid, 0·1 grm. pepsin must dissolve 0·3 grm. dry albumin from 100 grms. of a solution calculated to contain 1 per cent. dried residue, and made from dry albumin, in two hours at 55° C. The estimation is carried out as follows: In each of five 200 c.c. conical flasks is placed 100 c.c. of a clear filtered 1 per cent. albumin solution; 10 c.c. of $\frac{N}{1}$ hydrochloric acid are added to each. Two flasks serve as controls, and are heated for fifteen minutes in a vigorously boiling water-bath after addition of 10 c.c. of saturated sodium chloride solution; when cold, the coagulated mass is collected on a dried, weighed filter, freed from chlorine by washing with water, pressed between filter-paper, and dried to constant weight. The mean of the two is taken as the total albumin. To the contents of the three other flasks, heated to 55° C., are added different quantities of a 3 per cent. solution of pepsin, previously dried over sulphuric acid and powdered; 0·1 grm. pepsin should not be exceeded. The flasks are shaken once, and kept for two hours at 55° C. exactly; after the addition of 10 c.c. of sodium chloride solution, they are heated for thirty minutes in a rapidly boiling water-bath. Their contents are then cooled, filtered, washed free from chlorine, pressed, and dried. The difference in weight from the control gives the dissolved albumin, which should be calculated to a 1 per cent. albumin solution. A curve showing the quantity of milk-sugar to be added to bring a sample of pepsin down to the standard is given. O. E. M.

Influence of Metals Present in Urine on the Velocity of the Reduction Tests for Dextrose. G. Woker and E. Belencki. (*Pflüger's Archiv. Physiol.*, 1913, **155**, 45-67; through *Chem. Zentralbl.*, 1914, I., 302-303.)—Nylander's test for dextrose is retarded by the presence of mercury in the urine; the effect is least with sugar concentrations of from 0·37 to 0·75 per cent., and is not such as would cause the presence of dextrose to be overlooked. Other tests are also affected, some being retarded and others accelerated. Urine appears to contain two substances, one accelerating the action of dextrose on methylene blue, bismuth subnitrate, and mercuric chloride, whilst the other retards the reaction with silver nitrate and alkaline copper sulphate. It is also possible that one substance only may be present, and that this substance has a different effect on the various reactions. In both cases the velocity of the reduction is a function of the concentration of the urine.

W. P. S.

ORGANIC ANALYSIS.

Analysis of Asphalts. H. Loebell. (*Chem. Zeit.*, 1914, **38**, 18-20.)—Referring to a method proposed by Marcusson (ANALYST, 1912, **37**, 407) for the differentiation of natural asphalts from artificial asphalts or petroleum-distillation by-products, the author points out that a practically identical method, based on the action of strong sulphuric acid upon the bitumen dissolved in petroleum spirit, was described six years previously by C. Richardson. Neither process, however, is capable of distinguishing satisfactorily between natural and artificial asphalts or mixtures of the two, because the range of variation in the oily contents is far too wide. Still less is Marcusson's method available for the valuation of asphalt for building purposes, since the qualities required of a building asphalt stand in no relation to the determinations made. With regard to the percentage of solid paraffins as a basis of diagnosis, the author shows that his contentions are confirmed by analyses published by Scheller (*Petroleum*, 1913, 730) of a number of samples of Roumanian petroleum distillates. The solid paraffins in the portions distilling above 300° C. ranged from 13·92 to 0·41 per cent., and a number of the samples showed below 1 per cent., which is given by Marcusson as the limit for the paraffin-contents of pure natural asphalts. Scheller also shows that between petroleum asphalts poor and rich in paraffins there exist far greater differences than between petroleum asphalts poor in paraffins and natural asphalts. Petroleum products rich in paraffins are either worked for the recovery of paraffin wax or employed for some purpose where the high paraffin-contents are desirable; for the asphalt industry the products poor in paraffins are preferred. J. F. B.

Estimation of Carbon by the Wet Way. Simultaneous Estimation of Halogens. F. H. Thies. (*Chem. Zeit.*, 1914, **38**, 115.)—In continuation of his work (*Chem. Zeit.*, 1912, **36**, 917, and "Über die Bestimmung der fortschreitenden Oxydation mittels Chromsäure in Zellulose": Theissing, Münster i. W., 1913) the author considers the absorption of carbon dioxide and of halogens by calcium oxide. From the oxidation mixture of chromic acid and sulphuric acid, chlorine and bromine are evolved quantitatively, even in presence of silver, while iodine forms silver iodide. The gases leaving the oxidation-vessel are passed though a heated 30 cm. potash glass tube of 6 mm. bore, packed for half its length loosely, but continuously, with quicklime between tight-fitting asbestos stoppers. To this is attached a weighed soda-lime tube, protected by a second unweighed soda-lime tube. The reaction is stopped at the end of forty to fifty minutes. The increase in weight of the soda-lime tube gives the carbon dioxide not absorbed by the lime. The asbestos stopper nearest the soda-lime tube and the lime are transferred to a Bunsen carbon-dioxide apparatus; the rest of the carbon dioxide is determined by driving it off with nitric acid. The halogen is estimated in the nitric acid solution. Satisfactory results are given for a number of halogen compounds. O. E. M.

Difference between Calculated and Determined Calorific Values of Coal Gas. G. Weyman. (*J. Soc. Chem. Ind.*, 1914, **33**, 11-12.)—When the estimation of benzene and its homologues by absorption in alcohol is omitted, the calculated heat value of coal gas is always less than the value obtained by calorimeter. The difference between the two values is fairly constant for any one works provided no great change is made in the method of working. Changes made in the author's works showed, however, that the difference was far from being a constant, and he was led to make it a practice always to determine benzene by the well-known alcohol method before proceeding to the determination of the other constituents of the gas, a new departure in his laboratory. The result, when the gases absorbed by alcohol were calculated as benzene, was a very close agreement in every case between the calculated and determined calorific value. Incidentally a technical fault was detected, and the author lays stress on the value of the determination of benzene hydrocarbons, a subject which he says is overlooked in some recent textbooks, whilst one is cited as being definitely misleading. G. C. J.

Determination of Humus in Heavy Clay Soils. W. Beam. (*Cairo Sci. J.*, 1913, **7**, 219-224.)—The author refers to his previous work (ANALYST, 1912, **37**, 588), in which he recommends the removal of the hydrochloric acid by means of a cold solution of carbon dioxide, the filter being a Buchner funnel packed with a layer of asbestos and a supporting disc of paper; repeated extractions with ammonia were stated to be necessary, and it was found that the removal of clay from suspension in humus solution is readily accomplished by ammonium carbonate as suggested by Rather (ANALYST, 1911, **36**, 547), but the clay so precipitated carries down with it a portion of the humus. It is now found that the strength of the hydrochloric acid used for the extraction of the magnesium and calcium has a marked effect on the rapidity of the subsequent filtration, the stronger the acid the slower being the filtration; and it is suggested that the acid should not exceed a strength of 1 c.c. of strong acid diluted to 200 c.c. with water, a further improvement being the substitution of very dilute hydrochloric acid (one part of the above-mentioned 0·5 per cent. acid diluted with twenty-five parts of water) for the solution of carbonic acid, by means of which all classes of soils may be treated with ease and certainty. The clay is thus kept in a finely flocculated condition and the ammonia extraction is much more easily effected than when carbonic acid solution is employed. H. F. E. H.

New Indicator for Use in Determining End-Point in Titrations with Fehling's Solution. O. Carletti. (*Boll. Chim. Farm.*, 1913, **52**, 747-748; through *Chem. Zentralbl.*, 1914, I., 190.)—The method depends on the fact that cuprous cyanide is without influence on phenolphthalin, whereas the slightest trace of cupric salt instantly oxidises a portion of it to phenolphthaleïn, with production of the characteristic red colour in presence of alkali hydroxide. The reagent is prepared by boiling 1 grm. phenolphthaleïn, 10 grms. potassium hydroxide, and 100 c.c. water, with 5 grms. zinc dust, and filtering when colourless. It remains colourless sufficiently long if preserved in yellow glass bottles. When the titration approaches completion, a drop of the liquid is withdrawn from time to time, mixed in a basin with a drop of the indicator and 2 to 3 drops of a 10 per cent. solution of potassium cyanide. So long as cupric salts are present a red coloration is obtained. The method will detect 0·05 c.c. of unreduced Fehling's solution in 50 c.c. G. C. J.

Estimation of Lactic Acid in Liquids containing Protein. M. Oppenheimer. (*Zeitsch. Physiol. Chem.*, 1914, **89**, 39-45.)—Proteins are removed by Schenck's method (Abderhalden's "Handbuch der Biochemisch. Arbeitsmethoden," Bd. 2, S. 1 and 4), and the lactic acid estimated according to the method of v. Fürth-Charnass (ANALYST, 1910, **35**, 445) after previous extraction with ether.

E. W.

Influence of Ammonium Sulphate on the Rotation of Lactose. R. Roseman. (*Zeitsch. Physiol. Chem.*, 1914, **89**, 133-141.)—Tables are given showing the effect of varying quantities of ammonium sulphate on the specific rotatory power of lactose. On saturation with ammonium sulphate, the $[\alpha]_D$ falls from 52·53 to 50·47. In Salkowski's process for the estimation of lactose the liquid used for measuring rotation contains about 40 per cent. ammonium sulphate; this reduces the $[\alpha]_D$ from 52·53 to 51·55. The application of the correction suggested by Jahnson-Blom is theoretically justified, but in practice is better omitted, since the methods of

Kretschmer and Salkowski (Analyst, 1912, **37**, 350; 1913, **38**, 418) usually give higher results than other methods, and it is conceivable that this may be due to some unknown factor which compensates for the error due to the presence of ammonium sulphate. E. W.

Note on Methoxyl Estimations. R. J. Manning and M. Merenstein. (*Ber.*, 1913, **46**, 3983-3984.)—For the analysis of methoxyl compounds which yield low results by the ordinary Zeisel method, Herzig has recommended the addition of acetic anhydride and longer heating. By this addition and heating on three successive days for one and a half hours each day, the authors obtained the balance of the methoxyl sought for. They found, however, by blank experiments, that acetic anhydride and hydriodic acid alone yield appreciable quantities of silver iodide under these conditions, so that the method is not reliable. The addition of phenol on long heating also introduces a similar error, and cannot be recommended. J. F. B.

New or Little-Known Oil Seeds and Oils. (*Bull. Imp. Inst.*, 1913, **11**, 559-574.)—Among the oils examined were the following seed-oils:

	Sp. Gr. at 15·5/15·5° C.	Solid.-Pt. of Fatty Acids.	Acid Value.	Sapon. Value.	Iodine Value.	Hehner Value.	Soluble Volatile Acids.	Unsap. Matter.
		° C.					Per Cent.	Per Cent.
Amoora Rohituka (Bengal)	0·931	32·4	24·7	192·3	131·7	92·4	1·2	1·4
Eruca sativa (India)	0·915	2·4	—	175·7	101·6	—	—	—
Calophyllum inophyllum (India). Two samples	0·950	36·3	45·9	192·8	93·1	94·3	0·5	1·4
	—	—	77·5	194·9	97·7	—	—	—
Calophyllum inophyllum (Fiji)	0·951	—	65·5	199·5 (approx.)	96·2	—	—	—
Mesua ferrea (India)	0·932 (at 100°/15·5° C.)	30·5	16·2	204·9	92·2	91·9	6·7	1·4
Bassia latifolia (Mauritius)	0·862 (at 100°/15·5° C.)	43·2 to 46·0	—	188·3 to 195·3	56·7 to 61·5	94·2 to 96·4	0·2 to 0·9	2·0 to 3·5
Pentadesma butyracea (West Africa)	0·857	—	3·1	186·0	46·5	—	—	—
Acrocomia sclerocarpa (West Indies). Two samples	0·868	20·5	1·5	255·1	21·0	89·5	6·8	0·46
	0·867 (at 100°/15·5° C.)	—	1·3	253·7	16·2	88·5	5·7	0·4

C. A. M.

Volumetric Estimation of Nitro-Compounds and Nitroso-Compounds by Means of Titanium Chloride. H. Salvaterra. (*Chem. Zeit.*, 1914, **38**, 90.)—The method of Knecht (*J. Soc. Dyers and Colourists*, 1903, **19**, 169; 1905, **21**, 111, 292), which involves back-titration with iron alum solution and the use of ammonium thiocyanate as indicator, was found to give an indistinct end-point. To avoid this and other defects, the solution of nitro-compound or nitroso-compound is treated in an atmosphere of carbon dioxide with a measured quantity of standard titanium chloride solution; a measured excess of standard methylene blue solution is run in,

and the titration completed hot with titanium chloride. A sharp colour-change from green to light yellowish-brown or yellow marks the end-point. A tube, bent twice at right angles and drawn to a point, is substituted for the usual burette jet; the heating of the titration flask can then be carried out without affecting the contents of the burette.
O. E. M.

Estimation of Oxygen in Organic Compounds. M. C. Boswell. (*J. Amer. Chem. Soc.*, 1914, **36**, 127-132.)—The method previously described (ANALYST, 1913, **38**, 216) gives satisfactory results with substances containing 30 per cent. or more oxygen; but the results tend to be high when the percentage of oxygen is small, and in some cases the error may amount to several per cent. on the substance. A more elaborate method is now described, which makes it possible to estimate 10 per cent. of oxygen with an error not exceeding 0·3 per cent. on the substance. The method consists in heating the substance in a current of nitrogen, causing the vapours to pass over a small quantity of heated copper oxide of known oxidising value distributed on asbestos, weighing the water and carbon dioxide formed, and determining the oxidising value of the copper oxide remaining unreduced. The combustion tube of quartz, 1 metre long, is filled for a length of 60 cm. (starting 10 cm. from the forward end) with the prepared asbestos. This is prepared by dissolving about 4 grms. of copper in nitric acid, mixing in the asbestos, evaporating to dryness, and igniting. It is found that if this coppered asbestos be once reduced in hydrogen and then reoxidised in a current of oxygen for forty-five minutes, these processes may be repeated indefinitely with the production of a substantially constant amount of water during reduction, although neither the oxidation nor the reduction is quantitative. The substance is placed in a boat in the back part of the combustion tube, a rapid stream of pure nitrogen is passed through the tube and attached tubes for the absorption of water and carbon dioxide until air is expelled, the current of gas is then reduced to about one bubble per second, the layer of asbestos is raised to the highest temperature attainable in an ordinary combustion furnace with the iron trough removed, and the substance is heated, cautiously at first, and finally, after about twenty minutes, to a white heat. The tube is then allowed to cool, nitrogen is replaced by air, and the absorption tubes are disconnected and replaced by a tube containing sulphuric acid on pumice. Hydrogen is now passed through the tube until air is expelled, and the forward part of the asbestos is heated. At first the hydrogen is oxidised so rapidly that none escapes. When bubbles once more begin to pass, a further length of the asbestos is heated, and so on, the whole process of reduction occupying thirty minutes. As the reduction is never complete, though the velocity of reduction is very slow after thirty minutes, this process, like the subsequent oxidation in preparation for another experiment, should always be conducted watch in hand. A preliminary experiment, made once for all for the particular filling of the tube, has given the total available oxygen of the copper oxide, a. If the oxygen in the water and carbon dioxide obtained during the combustion in nitrogen be represented by b and c and the oxygen in the water obtained during the reduction in hydrogen by d, then the oxygen in the substance taken for analysis is given by $b+c+d-a$.
G. C. J.

Properties of the Phthaleïns as Indicators. A. Thiel. (*Sitzungsber. Medizin-naturwiss. Ges. zu Münster*, Nov. 7, 1913; through *Chem. Zentralbl.*, 1914, I., 424.)—The value of an indicator for volumetric work depends upon its sensitiveness, its rate of change, and the position of its end-point in the acidity-series. This position is ascertained by means of a series of solutions, each of which is at a different stage of acidity, as measured by its concentration of $H^{\cdot}$ ions. A titration-curve can thus be drawn, correlating the concentration of $H^{\cdot}$ ions with transition of colour. The so-called "characteristic" of the indicator is the curve which represents the "heterochromic" portion of the entire quantity of indicator present as a function of the $H^{\cdot}$. This heterochromic portion is represented by the formula $\frac{I_{II}}{I_I + I_{II}}$, in which I_I and I_{II} designate the forms of the indicator which bear the two colours. From the equation $\frac{[H^{\cdot}][I_{II}]}{I_I} = K$, based upon the law of mass action, follows the relation:

$$\frac{I_{II}}{I_I + I_{II}} = \frac{\frac{K}{[H^{\cdot}]}}{1 + \frac{K}{[H^{\cdot}]}}$$

As far as the position of the subject can be reviewed, it seems impossible to speak of the exact parallel between the electrolytic and the chromatropic equilibrium. This is easily conceivable in view of the physiological processes, the complicated electrolytic character of the indicators investigated, the errors due to dissolved salts, and the effect of colloids. The characteristic of phenolphthaleïn is shown graphically and exhaustively discussed, and those of tetraiodo- and tetrabromophenolphthaleïn are similarly given, with an explanation of the special properties of phenoltetrachlorophthaleïn, dinitro-, and tetranitro-phenolphthaleïn and of α-naphtholphthaleïn, which has a transition precisely similar in character to that of phenoltetrachlorophthaleïn. Experiments on ethyl alcohol are quoted to demonstrate how the weakening of the intensity of the indicators runs parallel to the sensitiveness to excess of alkali. These investigations have an important bearing on clinical work, a method having been evolved by which the actual $H^{\cdot}$-concentration of any colourless or slightly coloured liquid can be determined very easily by a colorimetric or even a simple coloriscopic comparison. For details the original paper must be consulted. O. E. M.

Determination of the Softening-Point of Pitch. M. Klinger. (*Chem. Zeit.*, 1914, **38**, 63.)—The estimation of the softening-point of pitch by Krämer and Sarnow's method, if not carried out under exactly standard conditions, may lead to somewhat variable results. The author therefore prescribes the following procedure, which is a modification of Krämer and Sarnow's method, in order to make the manipulation more convenient and to standardise the method: Sufficient pitch to give a fluid layer about 7 mm. thick is melted in a small flat-bottomed beaker, which is suspended in an oil-bath at 150° C. A tube, 10 cm. in length and 6 mm. bore, with both ends ground level, is dipped to the bottom of the melted pitch and allowed to remain for one minute. The upper end of the tube is closed with the finger when the tube is withdrawn, and the end which is filled with pitch is rested upon a cold glass plate until the pitch has solidified. The outside of the tube is then cleaned, while inside there remains a layer approximately 5 mm. high. On the top of this are poured 5 grms. of mercury from a graduated pipette, and the tube thus prepared is suspended in a beaker containing water at 40° C., supported inside another beaker containing water at the same temperature. A thermometer is placed with its bulb close to the layer of pitch, and the temperature is raised with a moderate flame at the rate of 1° C. per minute. The temperature at which the mercury falls through the pitch is taken as the softening-point.

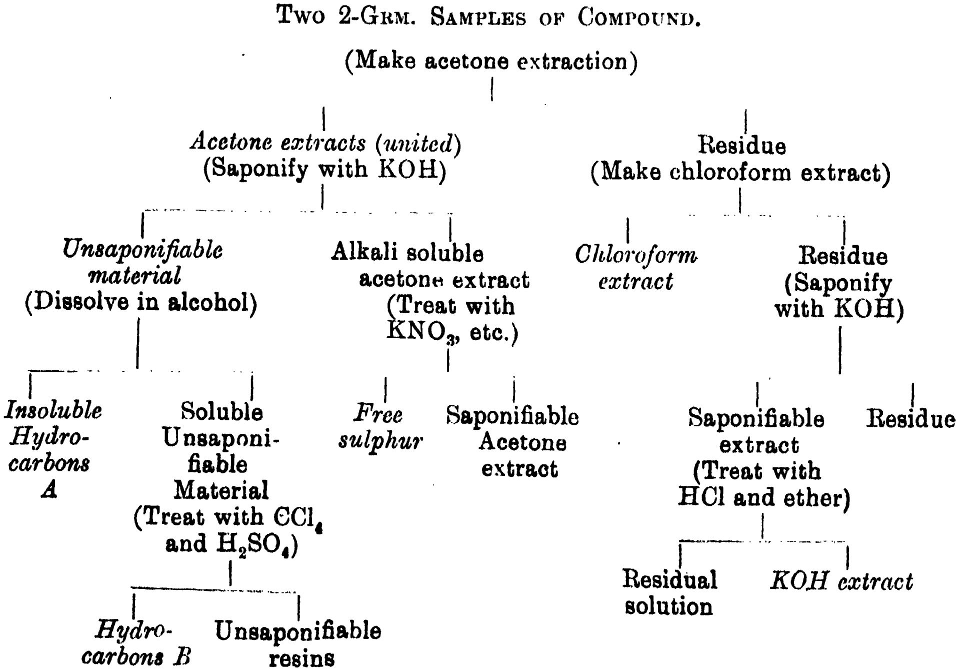
J. F. B.

Estimation of Pyruvic Acid. J. S. MacLean. (*Biochem. J.*, 1913, **7**, 611-616.)—The gravimetric estimation of pyruvic acid as phenyl-, nitrophenyl-, or asymmetrical diphenyl-hydrazone is inaccurate owing to the appreciable solubility of these hydrazones in water. More satisfactory results were obtained by estimating the quantity of phenylhydrazine removed during the reaction. The phenylhydrazine is estimated by decomposing with Fehling's solution at the ordinary temperature; after about half an hour the cuprous oxide is filtered off, dissolved in ferric sulphate solution, and estimated by titration with permanganate as in Bertrand's method. For the estimation of pyruvic acid the solution is treated with a measured quantity of a freshly prepared and colourless phenylhydrazine solution. After half an hour the hydrazone is filtered off, and the excess of phenylhydrazine in the filtrate estimated as described above. The presence of dextrose does not interfere with the estimation.

E. W.

Analytical Procedure for 30 Per Cent. Hevea Rubber Insulating Compound. (*J. Ind. and Eng. Chem.*, 1914, **6**, 75-82.)—In 1911 a conference of manufacturers and users of rubber insulation was held in New York, and, as a result, a representative committee was appointed to devise specifications and an analytical procedure for rubber insulating compounds. This committee has now issued a report, which includes a tentative specification for 30 per cent. Hevea rubber insulating compound, and also analytical methods, some of them new, which the committee think will serve to determine whether material delivered has in fact been compounded as specified. This report is issued to invite trial and criticism from outside the committee, which next year will make a final report embodying any useful improvements suggested meanwhile. The description of the analytical methods is very full, and no short abstract can take its place. The principle of the specification, however, may be stated. Only fine Para or smoked first latex Hevea rubber, sulphur, inorganic mineral matter, and refined solid paraffin or ceresin, may be used. The use of red lead and carbon is specifically forbidden. Upper limits are set to the proportions of waxy hydrocarbons, free sulphur, total sulphur (optional), unsaponifiable resins, chloroform extract, alcoholic potash extract, an upper and lower limit to the proportions of rubber and saponifiable acetone extract, and a lower limit to the specific gravity. The exact purpose of, and justification for, each of the limits are clearly set out—for example, the upper limit for rubber is designed to prevent the attainment of the specified electrical and mechanical strength by the use of an extra quantity of inferior rubber. Option is given to the purchaser to insert or omit the limit or total sulphur; with the limitation omitted, the use of sulphur-bearing fillers, which have some advantages, is possible, but the use of a notable proportion of reclaimed rubber is less certainly excluded. Probably, however, the other limits provide sufficient guard against the use of more than a small proportion of reclaimed rubber.

Some of the terms above used, such as "chloroform extract," "alcoholic potash extract," may be misleading without an outline of the analytical methods. Neglecting even important details and certain necessary corrections, these are as follows: Rubber is considered to be the difference between 100 and the sum of the waxy hydrocarbons, total sulphur, and fillers (free from sulphur) expressed as percentages. Total sulphur is determined in the usual manner. The other constituents are determined according to the following scheme:


TWO 2-GRM. SAMPLES OF COMPOUND.
(Make acetone extraction)
Acetone extracts (united)
(Saponify with KOH)
Residue
(Make chloroform extract)
Unsaponifiable material
(Dissolve in alcohol)
Alkali soluble acetone extract
(Treat with KNO_3, etc.)
Chloroform extract
Residue
(Saponify with KOH)
Insoluble Hydrocarbons A
Soluble Unsaponifiable Material
(Treat with CCl_4 and H_2SO_4)
Free sulphur
Saponifiable Acetone extract
Saponifiable extract
(Treat with HCl and ether)
Residue
Hydrocarbons B
Unsaponifiable resins
Residual solution
KOH extract

Substances shown in italics are weighed, others estimated by difference. The sum of *A* and *B* gives the total "waxy hydrocarbons." Of the constituents which figure in the specification, it will be seen that saponifiable acetone extract and unsaponifiable resins are determined by difference. In a mixing free from ceresin or paraffin, the unsaponifiable acetone extract, also a difference number, is identical with the unsaponifiable resins.

G. C. J.

Estimation of Caoutchouc in Crude Rubber. Pontio. (*Rev. gen. Chim. pur: et appl.*, 1913, **16**, 400-401; through *Chem. Zentralbl.*, 1914, I., 303.)—The process proposed by Marquis and Heim (ANALYST, 1913, **38**, 511) does not appear to be generally applicable since certain crude rubbers, particularly Para rubber, are only partially soluble in chloroform. It is recommended, therefore, that the crude rubber be dissolved by heating it with a suitable benzene homologue, the solution filtered while hot, and evaporated (finally *in vacuo*) to a syrupy consistence. The syrup is then poured slowly into alcohol or acetone, when the rubber is precipitated.

W. P. S.

Examination of Rosin Size. J. Marcusson. (*Chem. Rev. Fett Ind.*, 1914, **21**, 1-3.)—Commercial rosin sizes now contain numerous substances for which special tests are required. After estimating the water, insoluble impurities, alkali, and free and combined rosin, the sample should be treated with alcohol to separate substances such as glue, starch, dextrin, casein, gum, etc. A portion of the insoluble residue, which will contain weighting materials, such as barium sulphate, clay, etc., is ignited, and the ash estimated. An estimation is also made of the nitrogen, the absence of which shows that no glue, casein or albumin are present. Starch (detected by the iodine reaction) may be separated from dextrin and gum arabic by treatment with cold water, whilst gum arabic is precipitated by lead acetate from a cold aqueous solution. The difference in the optical rotations affords a further means of distinguishing between gum arabic and dextrin. Viscose may be recognised by its yielding hydrogen sulphide and cellulose hydrate on treatment with dilute acid. If the alcoholic precipitate is flocculent or thread-like and transparent, a vegetable gum may be present, and in that case an aqueous solution of the precipitate will give a gelatinous precipitate with lead acetate. The principal vegetable gums, including linseed gum and gum tragasol, may be distinguished from gum arabic by the fact that they yield precipitates with 5 per cent. tannin solution. If nitrogen is present in the alcoholic precipitate, special tests are made for animal sizes. Alcohol may precipitate dextrin and gum arabic simultaneously with nitrogenous substances. To detect them, the precipitate is dissolved in water, and the solution treated with an excess of tannin, and filtered. The filtrate is evaporated, and the residue taken up with water and again filtered. On adding excess of alcohol to the second filtrate, dextrin and gum arabic will be precipitated, and may be separated and identified as described. As a rule the alcohol-soluble part of a rosin size consists only of rosin and sodium resinate.

C. A. M.

Examination of Bleached Shellac. H. Wolff. (*Chem. Rev. Fett Ind.*, 1914, **21**, 4-5.)—Estimation of the ethereal extract affords a criterion of the purity of shellac. Thus, in the case of sixty-five samples of various origin, the ethereal extract ranged from 11·5 to 14·5 per cent. (with four exceptions), whilst thirty-nine samples gave values between 12·5 and 13·5 per cent. Still closer were the variations obtained by extracting the sample with benzene after previous extractions with petroleum spirit and ether. Thus, all the samples examined gave benzene extracts of 4 to 5 per cent., whilst in forty-five cases the extract ranged from 4·3 to 4·6 per cent. In estimating this value the powdered sample is ground up with twice its weight of sand, and the mixture successively extracted (preferably in a linen bag) with the three solvents, each extraction being continued for three to six hours. After each extraction the mass is ground up in a mortar with a small quantity of the next solvent and sufficient sand to yield a dry powder. It is advisable to dry the shellac at a temperature not exceeding 40° C. to expel water prior to the first extraction. The method cannot be used for the quantitative estimation of colophony.

C. A. M.

Estimation of Free Alkali in Soaps. E. Bosshard and W. Huggenberg. (*Zeitsch. angew. Chem.*, 1914, **27**, 11-20.)—The following method is proposed for the estimation of free alkali hydroxide and carbonate in soaps: 5 grms. of the sample are dissolved in 100 c.c. of neutral 50 per cent. alcohol, the mixture being heated under a reflux condenser to aid solution; after cooling, 20 c.c. of 10 per cent. barium chloride solution and 5 c.c. of 0·04 per cent. α-naphtholphthaleïn solution are added, and the mixture is titrated with $\frac{N}{40}$ stearic acid solution (in alcohol). This titration gives the amount of free alkali present as hydroxide. Should the soap also contain free alkali carbonate, a further 5 grms. of the sample are dissolved in 100 c.c. of 50 per cent. alcohol and the solution is titrated directly, after cooling, with the stearic acid solution, using phenolphthaleïn or α-naphtholphthaleïn as indicator. The difference between the two titrations gives the alkali present as carbonate. A correction of +0·01 per cent. must be applied when the free alkali amounts to 0·1 per cent., and of +0·04 per cent. when the alkali exceeds 0·4 per cent.

W. P. S.

Estimation of Total Sulphur in Commercial Benzene. K. Schenk. (*Chem. Zeit.*, 1914, **38**, 83-84.)—For one or other of several reasons, one of the most important being the small proportion of sulphur to be determined, several methods for the determination of sulphur in organic materials are unsuitable for its determination in benzene. In principle, Irwin's method (*J. Soc. Chem. Ind.*, 1901, **20**, 440) is a sound one, but it has practical disadvantages, and, as its author admitted, does not give exact results. Irwin mixed the benzene with ten volumes of alcohol (to reduce the tendency to a smoky flame) and burned this mixture in a lamp under the trumpet tube of the Referees' sulphur apparatus. The large amount of spirit used makes the method tedious, and as the spirit tends to vaporise first, there is always smokiness towards the end, and some of the mixture, of unknown composition, remains unconsumed at the end of the experiment.

The author vaporises the benzene in a current of air and burns the mixture under the wide cylinder of a Drehschmidt apparatus for the estimation of sulphur in gas, the products of combustion being sucked through three wash-bottles, each containing 20 c.c. of 1·5 per cent. hydrogen peroxide. At the conclusion of the combustion, the contents of the wash-bottles are titrated with sodium hydroxide. The vaporising apparatus, which is made by the Vereinigte Fabriken für

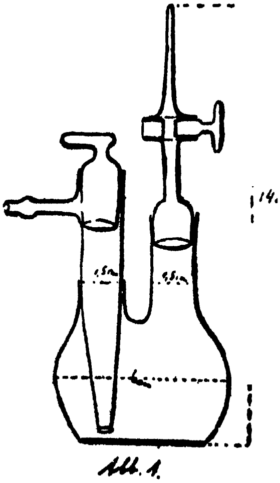


Abb. 1.

Laboratoriumsbedarf, Berlin, is illustrated. It is loosely filled with cotton-wool, tared, charged with 10 c.c. of benzene, reweighed, and connected by the horizontal inlet tube to a supply of air under slight pressure. The apparatus is set in a water-bath at 30-35° C. under the wide tube of the Drehschmidt apparatus, and the gas escaping from the vertical outlet tube is lighted. This outlet tube is a capillary of such dimensions as to exclude risk of explosion. The flame is kept about 0·6 cm. high and smokeless by regulating the air-supply and raising the temperature of the water-bath gradually to 75° C. The combustion occupies about six hours. Proportions of sulphur up to 1·25 per cent. can be determined with an error not exceeding 0·03 per cent. on the benzene. G. C. J.

Estimation of Water in Solid and Liquid Fuels by Distillation with Xylene. P. Schläpfer. (*Zeitsch. angew. Chem.*, 1914, **27**, 52-56.)—The principle of this method was approved by Huntly and Coste in their review of the whole question of the determination of water (Analyst, 1913, **38**, 174), and this particular modification of it, which, though unpublished, had been communicated to them as members of the International Committee on Coal Analysis, was briefly described by them and characterised as promising.

The principle of the method is old enough to be familiar to everyone, and is almost sufficiently stated in the title of this paper. The water in the distillate is measured, and the novelty embodied in the present paper consists of means and suggestions for making this measurement exact and for correcting the result for the mutual solubilities of water and zylene and other unavoidable sources of error. From 30 to 100 grms. of the fuel (according to its moisture-content) is distilled with 200 c.c. of commercial zylene, saturated with water, but freed from emulsified globules of water, if necessary, by warming until clear and then allowing to come to room temperature and decanting from any separated water. The distillate (150 c.c.) passes down a vertical condenser and is collected in a receiver 40 mm. wide in its upper portion, but only 6 mm. wide in its lower graduated portion, the constriction to the narrower diameter not being too sudden. A difficulty common to all methods in which one of two immiscible layers of liquid has to be measured is the persistence with which globules of the heavier liquid attach themselves to the glass above the meniscus dividing the two liquids. Zylene exhibits no tendency to get included in the aqueous layer provided the measuring tube is not narrower than 6 mm., and it is now found that the tendency for water to stick to the glass above the meniscus dividing the liquids may be wholly overcome by alternate steaming of the measuring tubes and treating them with chromic acid solution before each experiment. The zylene layer always contains emulsified water, but this is readily caused to separate by immersing the measuring tube for fifteen minutes in warm water. Notes are appended on the calibration of the measuring tubes and on the determination of the error due to distortion of the water meniscus by the superimposed layer of zylene. A more important error is due to actual loss of water. That there is unavoidable loss was shown by Michel (Analyst, 1913, **38**, 207), who stated that it was directly proportional to the water brought to measurement. The author proves beyond doubt that, with his apparatus at least, the relation is less simple than this. His curves and tables connecting amount of water measured with correction to be made, may be omitted here, as it is clear that every user of the method must determine these corrections for his own apparatus; but it may be said that the correction amounts to about 0·2 c.c. when 10 c.c. water are collected, and to 0·07 c.c. when only 0·28 c.c. is collected.

With the exception that certain graphite-like anthracites fail to yield more than 80 per cent. of their water, the method is shown to be more accurate than the toluene oven method, if relative accuracy be taken to mean degree of approximation of the results to those obtained on heating in a current of nitrogen and absorbing and weighing the water evolved. G. C. J.

INORGANIC ANALYSIS.

Detection of Traces of Aluminium. G. H. Petit. (*J. Pharm. Chim.*, 1914, **9**, 66.)—The iron, chromium, and aluminium are separated as hydroxides from the other metals in the usual manner; the precipitate is washed, to remove ammonium salts, and heated to boiling, but not boiled, with water and 2 grms. of pure crystallised barium hydroxide. On filtration, the iron and chromium hydroxides are removed unchanged; the filtrate contains the aluminium as barium aluminate. If aluminium is present in sufficient quantity, an immediate precipitate of aluminium hydroxide is obtained on acidulating the filtrate with hydrochloric acid and adding ammonia and calcium chloride and allowing to stand; a further precipitate of barium carbonate is slowly formed by absorption of carbon dioxide, and would mask the alumina if it were present only as a trace. In that case the filtrate is acidulated with a slight excess of sulphuric acid, the liquid concentrated to 20 or 30 c.c., the barium sulphate removed by filtration, ammonia added drop by drop in a maximum excess of 5 drops, and the liquid heated to boiling; on standing, the precipitate of aluminium hydroxide is formed. The method is sensitive to 0·001 grm. of aluminium in presence of chromium and iron in not too large an excess; with 5 grms. of iron 0·005 grm. may be detected. O. E. M.

Separation of Calcium from Magnesium. F. Halla. (*Chem. Zeit.*, 1914, **38**, 100.)—Although calcium and magnesium, when present in approximately equal quantity, cannot be separated by a single precipitation of the calcium as oxalate, small quantities of calcium can be separated from large quantities of magnesium with sufficient exactness if the precipitation be conducted as follows: To the boiling neutral solution solid ammonium oxalate is added until the precipitate, at first bulky due to magnesium oxalate, no longer appears to become less in amount. After standing several hours the precipitate is filtered off, and washed at first with hot water, but finally with cold water to counteract any tendency to form a colloidal sol when the concentration of soluble salts becomes very low. Even from solutions containing 1 grm. of calcium chloride in 50 c.c., 10 mgrms. magnesium chloride can be determined with an error not exceeding 0·3 mgrm., whilst 100 mgrms. can be determined with an error not exceeding 2 mgrms. In more dilute solutions the separation is said to be even better, but no test numbers are given to show the improvement.

When the ratio of magnesium to calcium is as low as 2 : 1, the error of the method becomes serious.

G. C. J.

Note by Abstractor.—The above is an abstract of the author's conclusions. His experimental results might be more compendiously expressed by the statement that the error of the method is 2 to 3 per cent., an error which is negligible when the proportion of calcium is very small.

Titration of Small Quantities of Carbonic Acid. A. Dorner. (*Zeitsch. Physiol. Chem.*, 1913, **88**, 425-430.)—The carbon dioxide is absorbed in $\frac{N}{100}$ barium hydroxide, and the excess of baryta estimated by titration with hydrochloric acid, using phenolphthaleïn as indicator. The removal of the barium carbonate is not necessary, for, although this substance has an alkaline reaction towards phenolphthaleïn, its reaction is suppressed by the presence of a sufficient amount of barium chloride. An absorption apparatus with burette connections is described and illustrated, in which provision is made for thorough absorption and for charging the apparatus and titrating without exposing the liquids to the laboratory atmosphere.

E. W.

Estimation of Chromium by Oxidation in Alkaline Solution. F. Bourion and A. Sénéchal. (*Comptes rend.*, 1913, **157**, 1528-1531.)—The method referred to is that in which sodium hydroxide is added to a solution of a chromium salt until the precipitate at first formed is redissolved, the solution is oxidised in the cold with hydrogen peroxide, of which the excess is destroyed by heating, a measured quantity of standard ferrous solution is added to the solution acidified with sulphuric acid, and the excess of ferrous solution is determined by titration with permanganate.

It is found that the method permits of the accurate determination of chromium in pure solution, and that iron does not interfere. On the contrary, the presence of iron is useful, as it reduces the time required to destroy the excess of hydrogen peroxide; sodium sulphate has a similar effect. Nickel, cobalt, and manganese, must be absent.

G. C. J.

Action of Sodium Hydrogen Carbonate Solutions on Difficultly Soluble Lead Salts. F. Auerbach and H. Pick. (*Arbeit. Kaiserl. Gesundheitsamte*, 1913, **45**, 191-196.)—When lead carbonate, lead chromate, or basic lead chromate are shaken for two days with dilute ($\frac{N}{10}$) sodium hydrogen carbonate solution at a temperature of 37° C., only from 0·3 mgrm. to 4·6 mgrms. of lead dissolves per litre of the solution. There is therefore no reason to suspect that these lead compounds, when taken internally, are rendered soluble by the pancreatic and intestinal fluids. The following process was employed for the estimation of the dissolved lead: 500 c.c. of the clear filtered solution were evaporated almost to dryness in a platinum basin. Sodium sulphide was then added, the mixture warmed slightly, and the lead sulphide collected on a filter. The sulphide was dissolved in boiling nitric acid, the solution evaporated in a porcelain basin, the residue dissolved in dilute acetic acid, and sodium chromate solution was added. After eighteen hours the lead chromate was collected, washed, dissolved in hydrochloric acid, carbon dioxide was passed into the vessel containing the solution, and potassium iodide was added, the liberated iodine being then titrated with thiosulphate solution. W. P. S.

Estimation of Iron in Iron Ores. E. Hintz. (*Zeitsch. angew. Chem.*, 1914, **27**, 9-11.)—The following was recommended by the Analytical Section of the Verein deutscher Chemiker, at the meeting held last year, as a standard method for the estimation of iron in iron ores: The powdered sample is placed in a conical heap on a sheet of paper, the heap is divided into two portions which are pressed flat and remixed; these operations are repeated twenty times. The last heap obtained is divided into four parts, and the two opposite quadrants respectively are collected separately. The two samples thus obtained are again well mixed and used for duplicate estimations. For the estimation itself, 5 grms. of each of the samples are placed in a 500 c.c. flask, moistened with 2 c.c. of water, and then digested for three hours at 60° C. with 100 c.c. of hydrochloric acid (sp. gr. 1·19). In the case of ores containing organic matter or pyrites, the portion taken for the estimation must be ignited in an open porcelain crucible before it is treated with hydrochloric acid. The mixture is then boiled, diluted with 150 c.c. of water, and filtered into a 500 c.c. flask. The insoluble residue is now ignited in a platinum crucible, fused with sodium hydrogen carbonate, and the fused mass dissolved in water and hydrochloric acid; the solution is oxidised, the iron precipitated with sodium hydroxide, dissolved in hydrochloric acid, and the solution added to the main solution. Instead of fusing with sodium hydrogen carbonate, the residue may be treated with hydrofluoric acid and sulphuric acid, and the iron then separated as described. In the case of ores containing chromium, the residue insoluble in hydrochloric acid is fused in a nickel crucible with a mixture of sodium peroxide and sodium hydroxide, the iron being then separated and added to the main solution. The acid solution containing the total iron is now diluted to 500 c.c., and 100 c.c. of it are evaporated to 50 c.c., and reduced while hot by the addition of concentrated stannous chloride solution, the latter being added drop by drop until the solution is colourless. Two drops are added in excess, the mixture is cooled, 25 c.c. of a 5 per cent. mercuric chloride solution are added, and after the lapse of at least two minutes the solution is rinsed into 2 litres of water treated previously with 80 c.c. of manganese sulphate solution (see below), and then rendered just pink with permanganate solution. The whole solution is now titrated with permanganate solution which has been standardised against pure ferric oxide. The permanganate solution should contain about 6 grms. of potassium permanganate per litre; the manganese sulphate solution is prepared by dissolving 200 grms. of crystallised manganese sulphate in 1 litre of water, and adding the solution to a mixture of 1,000 c.c. of phosphoric acid (sp. gr. 1·3), 600 c.c. of water, and 400 c.c. of sulphuric acid (sp. gr. 1·84). No decided evidence has been obtained as to the necessity of separating copper (when this is present in the ore) before the iron is estimated. W. P. S.

New Method of Estimating Mercury. C. Duccini. (*Gazz. Chim. Ital.*, 1913, **43**, 693-699.)—The method is based upon the reduction of the mercury salt by means of hydrazine sulphate and gravimetric estimation of the metallic mercury. The solution of the mercury salt is treated, with continual stirring, with an excess of a saturated solution of the reagent at the ordinary temperature, followed by 10 per cent. potassium hydroxide solution until the liquid is alkaline. After standing for six to eight hours the clear supernatant liquid is decanted, and the mercury is collected on a dry tared filter, and washed successively with water, 98 per cent. alcohol, and ether. It is then exposed for a few minutes to a current of dry air, left for five to six hours over calcium chloride in a desiccator, and weighed. The concentration of the solution of the mercury salt or the presence of free acid does not affect the results. In test estimations with solutions of mercuric chloride, mercuric cyanide, mercurous nitrate, and potassium mercury iodide, the mean error amounted to 0·10 to 0·13 per cent. of mercury on quantities of 0·25 to 0·30 grm. of the salts analysed.

C. A. M.

Gravimetric Estimation of Nitrites. N. Busvold. (*Chem. Zeit.*, 1914, **38**, 28.)—The nitrite is oxidised by means of silver bromate, and the reduced silver bromide estimated gravimetrically. From 1·4 to 1·5 grms. of silver bromate are dissolved in about 100 c.c. of water and 110 c.c. of 2 N acetic acid, heated to about 80° C. The solution of nitrite (about 1 grm. in 200 c.c.) is then added drop by drop from a burette, and is followed by 30 c.c. of sulphuric acid (1 : 4) at 85° C. After vigorous shaking and heating, the precipitate becomes bright yellow and the liquid clear. The precipitate is then collected in a Gooch crucible, washed with about a litre of hot water, dried at 130° C., and weighed. Parallel estimations showed that the method was accurate to within +0·1 per cent., whilst the accuracy of the permanganate method rarely exceeded +0·3 per cent. In the case of nitrites containing much chloride the method is much more accurate than the titration method.

C. A. M.

Loss of Noble Metals on Cupellation. J. Loevy. (*Chem. Zeit.*, 1914, **38**, 82-83.)—A detailed account of experiments, having for their object to determine the relative merits of French and German cupels, the use of French cupels, especially "Deleuil" cupels, being often specified, in spite of the publications of Rose (*J. Chem. Met. Soc. S. Africa*, 1905) and others to the effect that this preference is misplaced. Two French and four German brands were tried, but no English ones, though the author quotes Rose to the effect that English cupels, notably superior to "Deleuil" cupels, are now obtainable. Judged by the absorption of gold, a German brand (unnamed) was the best tried (absorption only 0·20 per cent.), the French brand "Velter" was second (0·22 per cent.), another German brand third (0·24 per cent.), whilst the "Deleuil" cupels and the other two German brands showed absorptions of 0·35 to 0·37 per cent. The test cupellations were made with about 80 mgrms. gold and 5 grms. lead, and the above figures represent averages of six determinations. Individual results, even with the best brand, exhibit wide differences from the mean (0·10 to 0·22), thus confirming Rose's statement that one must not assume a constant loss with a particular brand or even a particular consignment, but must test them repeatedly. Experiments with silver are also described. The order of merit with silver is not quite the same as with gold, but the silver results are clearly entitled to less weight on account of the impossibility of eliminating entirely loss by volatilisation or keeping this loss quite constant. G. C. J.

Dimethyl Phosphates of the Rare Earths (Fractionation of Rare Earths).

J. C. Morgan and C. James. (*J. Amer. Chem. Soc.*, 1914, **36**, 10-16.)—The solubilities of the dimethyl phosphates of the rare earths differ somewhat widely, so that mixtures of the earths may be resolved by fractional crystallisation of the dimethyl phosphates. An added advantage is the fact that, with the exception of lanthanum dimethyl phosphate, which is equally soluble in hot and cold water, the dimethyl phosphates of the rare earths are much less soluble in hot than in cold water. This circumstance makes it possible to effect a high degree of fractionation in a very short time, samples of gadolinium and of samarium having been prepared free from even spectroscopic traces of any other metal in forty-eight hours. Some of the solubilities of the dimethyl phosphates are appended:

Element.	Solubility at 25° C. Grms. per 100 c.c.	Solubility at 95° C. Grms. per 100 c.c.
Lanthanum	103·7	103·7
Cerium	79·6	65·0
Praseodymium	64·1	—
Neodymium	56·1	22·3
Samarium	35·2	10·8
Gadolinium	23·0	6·7
Yttrium	2·8	—
Erbium	1·78	—
Ytterbium	1·20	0·25

G. C. J.

Solubility of Mineral Phosphates in Citric Acid. G. S. Robertson.

(*J. Soc. Chem. Ind.*, 1914, **33**, 9-11.)—Mineral phosphates, even when very finely ground, usually yield only a small amount of soluble phosphate when extracted for thirty minutes with 100 parts of 2 per cent. citric acid solution. Yet it has been claimed that such finely ground mineral phosphate may have a manurial value equal to basic slag or bone meal, and field experiments are described in this paper which support this view. An explanation of these results was sought, and finally a series of successive extractions seemed to supply the key to the problem. In one case a sample of Tunisian phosphate was extracted in the usual manner, and yielded soluble phosphate equivalent to 8 per cent. tricalcic phosphate and 17 per cent. soluble lime. On re-extraction for thirty minutes with fresh citric acid solution, 17 per cent. of tricalcic phosphate went into solution, doubtless because the excess of lime that had largely neutralised the first liquor had been removed. A third extraction yielded another 13 per cent. of soluble phosphate, and after five extractions the whole of the tricalcic phosphate (49 per cent.) had been dissolved. The author considers that these results demonstrate the worthlessness of the citric acid test as a means of judging the relative value to the plant of phosphatic manures. The original purpose of the citric acid test was to detect adulterations, against which purpose nothing is urged. It is the subsequent interpretations which have been put on the results of this test that the author considers unjustified. G. C. J.

Analysis of "Silicol" and Other Silicon Compounds used for the Production of Hydrogen. G. F. Jaubert. (*Rev. gén. Chim. pure et appl.*, 1913, **16**, 341; through *Chem. Zentralbl.*, 1914, I., 74.)—Fifty grms. of the substance are weighed out and divided equally between five cartridges of smooth paper, closed at one end, and pinched together at the other. The cartridges are introduced singly into a 2-litre evolution-flask, and the stopper immediately replaced. The flask communicates with a series of three wash-bottles, containing respectively water, to arrest steam; bromine water and bromine, to arrest hydrogen phosphides; and 10 per cent. sodium hydroxide solution. The last wash-bottle is provided with a thermometer not dipping into the liquid, and is connected with a gas-meter. The evolution of gas from one cartridge being finished, another is put into the flask, until all five have been introduced. The gas volume is corrected for temperature, as measured by the thermometer, and for barometric pressure. The phosphorus is estimated in the contents of the second wash-bottle, after removal of the bromine by boiling, in the usual manner.

The gravimetric estimation of silicol is carried out as follows: (1) *Silicon.*—In a nickel dish 8 cms. in diameter 0·5 grm. of the substance is carefully heated with 5 grms. of dried sodium carbonate and 10 grms. of sodium peroxide, finally with agitation, until a clear fused mass is obtained. This is dissolved in cold water, acidulated with hydrochloric acid, evaporated to dryness, treated with 10 c.c. of hydrochloric acid in 100 c.c. water, and filtered; the treatment is repeated upon the filtrate. The united precipitates are dried, ignited, and weighed; the silica is driven off with hydrofluoric and sulphuric acid, and the residue ignited and weighed. The loss gives the silicon as silica. (2) *Titanium.*—The residue is fused with 1 grm. of potassium hydrogen sulphate, the melt dissolved in a large quantity of hot water, and added to the filtrate from the silicon determination, the liquid neutralised with ammonia, treated with 1 to 2 drops of sulphuric acid, and 60 c.c. of sulphur dioxide solution, and boiled for one hour, the titanium oxide filtered off, ignited, and weighed. (3) *Iron.*—The filtrate is made up to 500 c.c., and 250 c.c. of this are acidulated with sulphuric acid and titrated with permanganate. (4) *Aluminium.*—To the remaining 250 c.c. of solution, neutralised with ammonia, are added 4 c.c. of hydrochloric acid, 2 grms. of tribasic ammonium phosphate dissolved in water, and 10 grms. sodium hyposulphite in water containing 15 c.c. of acetic acid. The liquid is boiled for fifteen minutes, and the precipitate washed with boiling water, ignited in a porcelain crucible, and weighed. A sample of silicol, thus analysed, gave 70·35 per cent. silicon, 11·90 iron, 3·55 titanium, and 14·05 aluminium. For the valuation of silicon compounds for the production of hydrogen, the gas-evolution method should always be used. From 50 grms. of silicol are obtained 70-75 litres of hydrogen. O. E. M.

Detection and Approximate Estimation of Traces of Thiosulphates in Sulphites. J. Bodnar. (*Chem. Zeit.*, 1914, **38**, 146-147.)—When a soluble sulphite is treated with silver nitrate solution, a white precipitate of silver sulphite is formed; but when the sulphite contains thiosulphate, the colour of the precipitate is yellow, orange, brown, or black, according to the quantity of thiosulphate present. This coloration is due to the formation of silver sulphide by the decomposition of the silver thiosulphate according to the equation: $Ag_2S_2O_3 + H_2O = Ag_2S + H_2SO_4$. The quantity of thiosulphate may be estimated by mixing 5 c.c. of a 10 per cent. solution of the sulphite with 10 c.c. of a 14 per cent. silver nitrate solution. The coloration obtained is then compared with those produced by similar quantities of pure sodium sulphite, to which have been added known amounts of thiosulphate. The test will detect 0·3 c.c. of $\frac{N}{50}$ thiosulphate solution in 5 c.c. of 10 per cent. sodium sulphite solution; this corresponds with 0·09 per cent. of sodium thiosulphate in crystallised sodium sulphite. If the mixtures are exposed to light for some time, the yellow coloration changes to grey, and the latter colour will then be noticed in a mixture containing only 0·1 c.c. of $\frac{N}{50}$ thiosulphate solution (equivalent to 0·032 per cent. of thiosulphate) in the sulphite. Pure silver sulphite turns slightly yellow after long exposure to light, but not to such an extent as does silver sulphite prepared from a sulphite containing 0·043 per cent. of thiosulphate. W. P. S.

Volumetric Estimation of Vanadium. G. Wegelin. (*Zeitsch. angew. Chem.*, 1914, **53**, 81-99.)—As a standard substance for the volumetric estimation of vanadium the pure pentoxide is employed, preferably prepared from the oxychloride by hydrolysis; if the highest accuracy is not required, the pentoxide may be obtained by the ignition of ammonium vanadate purified four times by re-crystallisation. The estimation of vanadium by permanganate, after reduction by sulphurous acid, may be performed with quantities of 0·3 to 0·4 grm. of vanadium pentoxide, with a maximum error of 0·2 per cent. For this purpose iron must be absent; the interfering influence of chlorides may be avoided by the addition of manganese sulphate. In presence of iron the permanganate method of Müller and Diefenthaler, after reduction with alcohol and hydrochloric acid, gives good results. The solution to be titrated is placed in an Erlenmeyer flask, having a diameter at the bottom of 10 cm., together with 20 c.c. of strong hydrochloric acid and 50 c.c. of alcohol. The mixture is heated on wire gauze, and evaporated to about 30 c.c., 5 c.c. more alcohol are added, and the whole is concentrated to about 5 c.c., care being taken not to allow the liquid to dry up on the sides of the flask. It is then diluted with water to 120 to 150 c.c., and titrated with $\frac{N}{10}$ permanganate to a colour permanent for half a minute. Before titration, if iron is absent, 6 c.c. of a solution containing per litre 70 grms. of crystallised manganese sulphate and 130 c.c. of concentrated sulphuric acid are added; in presence of iron the same solution with the addition of 138 grms. of phosphoric acid (sp. gr. 1·7) per litre is used. An alternative iodimetric method, due to the same authors, is also satisfactory if performed in the following manner: The neutral or slightly alkaline pentavalent vanadium solution (about 70 c.c.) is treated with 2·5 grms. of potassium ferrocyanide free from the ferri salt. After this is dissolved, 20 c.c. of $\frac{N}{2}$ sulphuric acid, 3 grms. of potassium iodide, and 20 c.c. of zinc sulphate (15 grms. of the crystallised salt per 100 c.c.) are added in the order named. After standing for ten minutes, the solution is diluted to 250 to 300 c.c., and the liberated iodine is titrated with thiosulphate. The results are as accurate as those obtained with permanganate, provided no interfering substances are present. J. F. B.

Applicability of Conductivity Methods in Examination of Natural Waters. A. Doroschevski and S. Dvorzanczyk.

(*J. Russ. Phys. Chem. Soc.*, 1913, **45**, 1489-1528; through *Chem. Zentralb.*, 1914, I., 183.)—The authors have applied conductivity methods to a variety of natural waters, the total solids of which were also determined directly by evaporation with a known weight of sodium carbonate, and drying at 110° C., the American official method, which they consider the best direct method. They calculate total solids from the conductivity by the formula $S = \frac{k_c . 10^6 . A}{C}$, where S is the total solids, k_c the observed conductivity, C the concentration of the water ($C = 1$ when it is unnecessary to dilute the water to bring the measurement within the range of the apparatus), and A is a constant which approximates 0·67. The exact value of A necessarily varies with the exact composition of the saline residue, and differs most widely from the above-given average value in the case of waters containing notable amounts of silicic acid or organic matter. A is, however, very nearly constant for many water sources, and once this fact has been established and the average value of A determined, conductivity measurements afford a more accurate as well as more rapid measure of the total solids than a direct determination in the usual manner. The authors estimate the error of the conductivity method at 2 per cent., that of a direct determination of solids at 5 per cent., provided this is made in the "usual manner." G. C. J.

Note.—The abstract in the *Chem. Zentralb.* does not define the "usual manner" in any way, but it is clear that the authors regard the American method as a distinct improvement on it.

APPARATUS, ETC.

Industrial Applications of Various Processes and Systems for the Measurement and Nomenclature of Colour Shades. P. Krais.

(*Zeitsch. angew. Chem.*, 1914, **27**, 25-37.)—The author makes a critical survey of the principles and practice of the various colour-measuring systems. No one of them is perfectly satisfactory; at the best only a compromise can be obtained. One of the first practical difficulties met with is the nature of the light; in the second place there arises the differences occasioned by the nature and reflection of the surface and the "top" shade. Systems based on transmitted light are not suitable for general application. Reviewing the literature of the subject, the author speaks highly of Rosenstiehl's work, and particularly his latest book, "Traité de la Couleur au point de vue Physique, Physiologique et Esthétique," Paris, 1913. Nine systems of practical colorimetry are critically described. Lovibond's system is credited with considerable practical value for many technical purposes, but the author has failed to find numerical coincidence with certain combinations. The difficulty with any transmitted light system is that hitherto it has been impossible to obtain a spectrally pure yellow light filter, and hence the usual three-colour reproduction processes are inaccurate as regards the yellow. As a textile colorist, the author has obtained the best practical results with Baumann's colour chart, with which he is able to match shades with great rapidity and ease. It is, however, capable of still finer development than is represented by its present form. The advantages of Baumann's system are: cheapness, ease of manipulation, large number of colour shades obtainable, publication of the colour recipes, and the wide scope of its utility. Disadvantages are: optical non-uniformity owing to the use of commercial pigments and a somewhat complicated nomenclature, which, however, may be substituted by numbers. The author considers that an absolutely matt, non-lustrous pigment surface must form the basis of any practical system, in preference to oil-printed colours or transparencies. The question of colour intensity is still very obscure, and in this connection Rosenstiehl's views give the best basis of compromise. J. F. B.

Dennstedt Combustion Furnace with Internal Electrical Heating.

C. Milchsack and W. A. Roth. (*Zeitsch. angew. Chem.*, 1914, **27**, 5-7.)—For the combustion analysis of organic compounds by Dennstedt's method the authors have designed a furnace electrically heated internally by means of platinum wires. The furnace comprises an ordinary hard-glass tube, 65 cms. long and 2 cms. in diameter (see Fig. 1), supported on an iron channel, which may be lined with asbestos and covered with asbestos-lined plates. At the point where the boat containing lead peroxide or other reagent is situated, a coil of nickel wire is embedded in the asbestos and heated electrically so that the temperature inside the tube reaches about 300° C. This is the only external heating in the whole apparatus. The contact-body consists of a glass cylinder, 6·5 cms. long and 1·5 cms. in diameter (see Fig. 2), closed at the front end, and joined at the other end to a glass tube, 35 cms. long and 0·7 cm. in diameter, which passes through the rubber stopper of the combustion tube. This

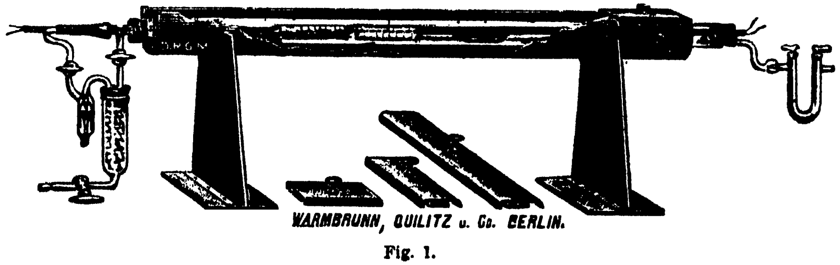


Fig. 1.

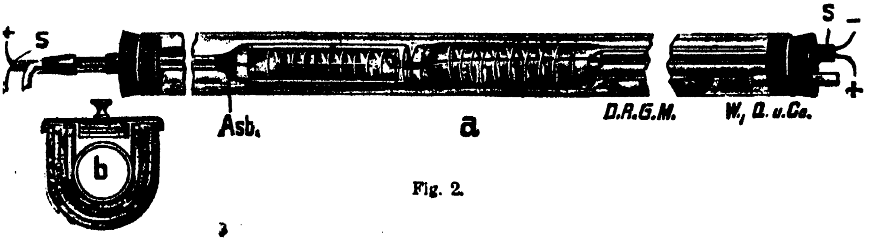


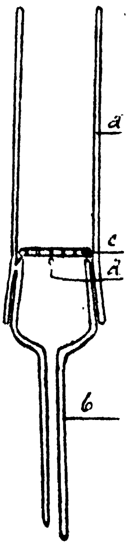
Fig. 2.

narrower tube serves to carry the copper wires conveying the current, which are soldered inside to the platinum wire spiral, about 0·5 mm. thick, constituting the active contact-substance. The platinum wire is fused into the walls of the glass cylinder at both ends, the spiral being carried along the outside of the cylinder and terminating in a projecting loop or "nose," which extends in front of the closed end of the cylinder to within a short distance from the boat containing the substance. This boat is carried in a tube about 8 cms. long, which is inserted in the gas-leading member of the apparatus and around which is wound a spiral of platinum wire, 0·4 mm. thick. The ends of the spiral are soldered to copper wires passing through the gas-leading tube, and the connections are made as shown in the figure. In starting a combustion, the nickel wire outside the lead-peroxide portion of the tube is first heated up by a current of about 6 ampères at 13 volts. Then the contact-spiral is gradually brought to a dull red glow with about 6 to 7 ampères at 9 volts. The heating of the other spiral will depend on the volatility of the substance; for the burning of charcoal about 6·5 ampères at 12 volts are required. Combustion starts at the point where the "nose" of the contact-spiral is assisting the work of the heating spiral, and proceeds slowly backwards under the control of the current, which is regulated by two rheostats. Combustions, even of the most difficult substances, can be completed in one to two hours and the results are perfectly satisfactory.

J. F. B.

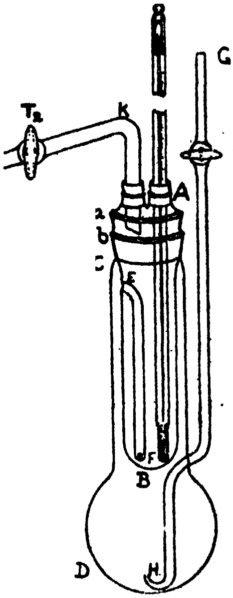
Filter from which a Precipitate may be Removed Quantitatively. E. Trutzer. (*Chem. Zeit.*, 1914, **38**, 55.)—The filter consists of two glass tubes, *a* and *b*, fitting into one another by a ground-in joint (see figure); a perforated porcelain plate *c* is supported on the upper edge of the lower tube, and a layer of asbestos is placed on the plate. When a precipitate has been collected on the filter and washed, the lower tube is removed, the upper tube is inverted over any suitable vessel, the plate is withdrawn by means of the small loop *d* attached to its lower side, and the precipitate, together with the asbestos, is rinsed into the vessel.

W. P. S.



Modified Lothar Meyer's Apparatus for the Purification of Mercury. F. Friedrichs. (*Zeitsch. angew. Chem.*, 1914, **27**, 24.)—As is well known, the purification of mercury in this apparatus is attained by allowing the metal to fall in the form of a fine stream of droplets through a long column of dilute nitric acid. In the modification now described a number of indentations are made in the opposite sides of the vertical tube containing the nitric acid, so that the droplets of mercury are deflected from side to side, and caused to take a longer path through the acid. The mercury is admitted to the top of a tube through a funnel, the stem of which is drawn out to a fine jet; if necessary, the mercury may be filtered previously through muslin.

W. P. S.



Improved Apparatus for the Determination of Molecular Weight by the Landsberger-Sakurai Method. W. E. S. Turner and C. T. Pollard. (*Chem. Soc. Proc.*, 1913, **29**, 349-352.)—The apparatus described is a modified form of that devised by Turner (*Chem. Soc. Trans.*, 1910, **93**, 1184). The chief alteration is the use of the boiler as the constant temperature jacket. When this is done, and the entrance for the vapour stream is high up in the molecular weight tube, the cooler solvent or solution in the tube is heated considerably before the entrance of vapour, and the amount of condensation is thus diminished, so enabling a longer series of consecutive readings to be obtained. The molecular-weight tube, *AB*, 17·5 cms. in length, 2·8 cms. diameter in the main portion, and 3·5 cms. at the mouth, carries a ground-glass stopper, with two tubulures fitting flush with the mouth of the tube at *a*. To the solvent or solution under measurement, vapour is admitted through two perforations at the bottom of the tube *EF*, the entrance *E* being 12 cms. from the lower end of the molecular-weight tube. The outer jacket *CD*, about 5 cm. in diameter, fits the molecular-weight tube at a second ground joint at *b*, and carries a safety tube, *GH*, provided with a tap, T_1. Whilst a determination is in progress T_1 is usually closed in order to drive a steady stream of vapour through *E*, but when the tube *AB* has been removed for weighing, a cork is inserted in the mouth of the boiler, and T_1 is opened to admit dry air drawn in through drying tubes connected at *G*. Hygroscopic solvents are thus protected. Vapour escapes by the tube *K*, of length as short as possible, connection with the condenser being made at a third ground joint, so that the molecular-weight tube, together with the thermometer and escape tube, can be lifted away bodily for weighing, during which process the tap T_2 is closed. It is advisable to protect the cylindrical portion of the outer jacket against loss of heat by radiation with a lagging of asbestos paper. Ethyl ether, carbon disulphide, and water, are easier solvents to deal with than alcohol, and with them a long series of readings is obtainable. When a solvent boiling over 100° C. is used, the number of readings becomes less; above 150° C. the apparatus can conveniently only be employed where it is sufficiently accurate to assume that the volume of the solution is the volume of the solvent, for in such a case it is unnecessary to detach the molecular-weight tube, thus avoiding handling it at a high temperature. Details requiring attention and necessary corrections were discussed in the previous paper (*loc. cit.*). H. F. E. H.

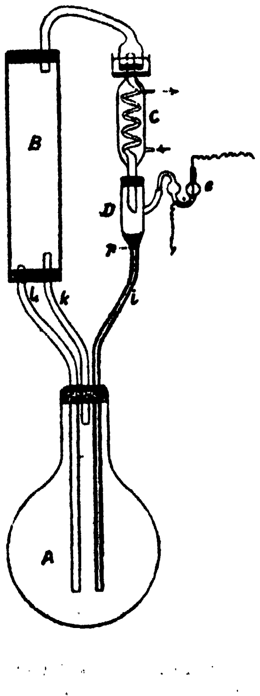
Steam Generator. J. A. Murray. (*Chem. News*, 1914, **109**, 29-30.)—The apparatus consists of a copper cylinder fitted with an air-tight lid, and with the bottom externally concave. The central tube, which is coiled, is brazed into the cylinder. The steam which passes through the coil becomes superheated without any increase of pressure, and may be used for different purposes, such as heating steam-baths, ovens, etc., to temperatures above 100° C., etc. Such baths may be made of zinc or tin in place of copper, with a consequent saving of expense. The apparatus itself can be made for about £1. At the end of the steam pipe a collar is brazed, into which fits tightly an angle tube, which can be turned in any desired direction and so be accommodated to different pieces of apparatus.

H. F. E. H.

Improved Absorption Tube for Spectroscopic Analysis. U. Müller. (*Chem. Zeit.*, 1914, **38**, 116.)—The absorption tube is ground at the ends and covered with flat glass disks cemented on. It is filled through a side tube at one end, which dips into the liquid to be examined, when suction is applied to a side tube pointing upwards at the other end. O. E. M.

Temperature of Sublimation. J. Joly. (*Phil. Mag.*, 1914, **27**, 1-14.)—By means of the apophorometer (see ANALYST, 1913, **38**, 302) the temperature of sublimation and the nature of the sublimate have been investigated for a large number of minerals containing Sb, As, S, Fe, Bi, Se, and Pb. Quantitative experiments upon several of these minerals are also described.

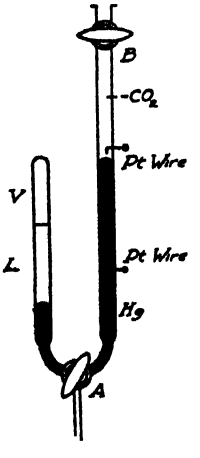
High Temperature Thermostat. A. Gerasimov. (*J. Russ. Phys. Chem. Soc.*, 1913, **45**, 1668-1677; through *Chem. Zentralbl.*, 1914, I., 94.)—There is no difficulty in maintaining a thermostat at a few definite temperatures corresponding to the boiling-points of certain liquids easily obtained in a state of purity. Some intermediate temperatures are more difficult to maintain, and the apparatus figured is designed to secure this end by employing mixtures of liquids of different boiling-point and insuring condensation and quantitative return to the boiler of all the vapours, so that there is no change of temperature in the thermostat once equilibrium has been obtained. The illustration, in which *B* is the thermostat, *A* the boiler, and *C* the condenser, is self-explanatory, except that *p* represents a piece of fine wire gauze to insure a regular chain of air bubbles and liquid drops in the tube *i*, and *e* is intended to control the gas supply to the burner below the boiler. The U-tube *e* contains mercury, and where the pressure within the apparatus exceeds a certain limit, this mercury moves and breaks an electrical circuit including the coils of an electromagnet, which normally keeps open a spring clip on the principal gas supply. G. C. J.



Calibrating Thermometers by Means of Floating Equilibrium. T. W. Richards and J. W. Shipley. (*J. Amer. Chem. Soc.*, 1914, **36**, 1-10.)—The authors have already described (ANALYST, 1912, **37**, 333) a method for the quantitative analysis of solutions by precise thermometry. It was pointed out in that paper that the method would serve equally well for the calibration of thermometers by means of solutions of known density or concentration as for the determination of concentrations by means of known temperature differences. The present paper describes experiments showing exactly how the phenomenon of floating equilibrium may be used to standardise thermometers. The method consists in determining the temperatures at which different analysed solutions of a given substance attain exactly the density of a given float, such as was described in the former paper. When the almost linear curve, showing the relation of this floating equilibrium-temperature to percentage composition, has once been established, it serves for the calibration of any thermometer at any time. The use of the method for determining the coefficient of expansion of liquids is also indicated. G. C. J.

Vacuum-Jacketed Thermometer. A. Hahn. (*Zeitsch. angew. Chem.*, 1914, **27**, 24.)—In order to avoid the necessity of making a correction for the difference in temperature between the bulb and the mercury thread when the whole of the latter is not immersed in the liquid of which the temperature has to be ascertained, the author describes a thermometer provided with a vacuum jacket extending from the top of the instrument to the upper part of the bulb. The lower portion of the jacket, between the bulb and the zero point, may be silvered. W. P. S.

Electrical Contact Vapour-Pressure Thermoregulator. A. L. Feild. (*J. Amer. Chem. Soc.*, 1914, **36**, 72-76.)—Ether (or other liquid) is charged into the left limb of the apparatus through the 3-way stopcock *A*, the whole apparatus being inverted for the purpose. Only enough ether is used to fill about one-third of the limb, and it is usually more convenient not to expel air, though this can readily be accomplished by heating the apparatus. With the apparatus still inverted, the cock *A* suitably turned, and cock *B* closed, mercury is introduced into the right limb. *A* is then turned so as to close both limbs of the apparatus, which is now placed upright, and *B* is opened. Connection is made between the two limbs, *B* is closed, and the apparatus placed in the thermostat it is intended to control. The thermostat being at the desired temperature, mercury is run off or added until it just makes contact with the upper platinum wire. The two platinum wires are connected with two dry batteries and a 150 ohm relay, which makes or breaks the circuit (or one of the circuits) supplying heat to the thermostat. The thermoregulator is less sensitive than a toluene thermoregulator can be made, but it is free from the lag which limits the practical utility of large toluene regulators in controlling the temperature of thermostats situated in ordinary rooms and exposed to wide variations of external temperature. As an example of the rapidity with which it responds, the following experiment is described: A regulator adjusted to 30° C. was removed from the bath, and allowed to attain the room-temperature of 25° C., whilst the bath was raised to 32·1 C. The regulator was then replaced, and within thirty seconds operated the



relay. By means of this apparatus, the temperature of a bath of 70 gallons capacity, situated in an ordinary laboratory, has been kept constant within 0·01 to 0·005 C.° for several days at a time. G. C. J.

New Viscosimeter. G. F. White and R. H. Twining. (*Amer. Chem. J.*, 1913, **50**, 380-389.)—In this preliminary paper is described a method for determining with accuracy the viscosity of undercooled liquids. Although water can be undercooled very little without spontaneously crystallising, especially if kept in motion, the authors have commenced their study with this substance in order to test the accuracy of the apparatus employed and for other reasons. The instrument is a modification of the form previously used by one of the authors (ANALYST, 1912, **37**, 143). The determinations were always made in duplicate by taking, first, the time of descent of the liquid from one point to another, and, secondly, the time of ascent with the pressure reversed. At 25° C., under a pressure of about 130 cms. of water at 20° C., about 415 seconds is required for the given volume to pass through the capillary, and at 0° C. about 815 seconds; below this temperature the viscosity of water increases very considerably, and a smaller volume is therefore used, thus reducing the time to about 315 seconds. Concordant results are obtained when thus varying the volumes. Values for water at temperatures ranging from 20° to −9·3° are recorded, the Poisseuille formula being employed as before for the calculation of the following results, which agree very closely with those of Bingham and White, Thorpe and Rodgers, Hosking and others:

Temperature.	Viscosity.
0°	0·01798
5°	0·01523
10°	0·01307
15°	0·01139
20°	0·01003

H. F. E. H.

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REVIEWS.

A TREATISE ON QUANTITATIVE INORGANIC ANALYSIS, WITH SPECIAL REFERENCE TO THE ANALYSIS OF CLAYS, SILICATES, AND RELATED MINERALS. By J. W. MELLOR, D.Sc. Pp. 778. London: Chas. Griffin and Co., Ltd. 1913. Price 30s. net.

This is the first of a two-volume treatise on the ceramic and silicate industries, but is an entirely independent work, and may be regarded, as its title indicates, as a general treatise on analysis. Even this somewhat wide definition of the scope of the work is incomplete, and the service which Dr. Mellor has done to the profession is such as imposes on the writer a duty very different from that of the ordinary reviewer.

Dr. Mellor indicates that his book has been written specially for students, and he could not have given a better description of what his intentions were, and of what he has succeeded in doing, if we include under the term "students" all workers in inorganic chemistry. One seldom reads books on analytical matters for the mere pleasure of reading them. They are naturally employed mainly as books of reference; but the present volume would be worth reading through, notwithstanding its length of nearly 800 pages, on account of the remarks which have been interpolated throughout to increase the interest of the description on general lines, and to emphasise those necessary precautions which not only have a special bearing on the actual matter being discussed, but are of general interest.

The writer feels it a duty to urge not only analysts, but all who are interested in chemical manipulation, to consult this work as one would ask advice from a friend who has full knowledge of the straightforward work entailed in general analysis, etc., and is also an enthusiast, with an intimate and critical experience of the difficulties which are constantly cropping up. Analytical work is both an art and a science, and the ordinary work of an analytical laboratory entails an amount of research work which few realise, and for which little credit is given. Dr. Mellor's work brings out this fact very emphatically. He would deserve the thanks of our profession for that alone, but the manner in which he has managed to clothe an ordinarily dry and somewhat uninteresting subject in a pleasing garb adds to our indebtedness to him.

The book commences with an interesting introduction dealing with the principles of analysis, and finishes with a series of tables and a list of books useful for the shelves of a laboratory. The book may be regarded as valuable to everyone who is engaged upon, or even interested in, the testing of both ordinary and out-of-the-way inorganic materials. For the worker on organic substances the volume is also full of valuable suggestions. It not only deals with methods of analysis, but covers, briefly and well, such points as the calibration of instruments, the testing of reagents, and (as footnotes) a most complete bibliography, generally in the form of references to original papers, instead of, as is so often done, to abstracts. The keynote of the whole work is common sense, and the author shows an intimate knowledge both of the methods to be used for determining the amounts of constituents known to be present, and—a far more difficult matter—the best way to look for them, and to avoid the pitfalls which the analyst is constantly finding in his path. The question of sampling, the use of sieves, the losses and changes produced in materials during grinding, etc., the attack on vessels by the reagents used, the storage of chemicals and of distilled water, simple methods of testing, abbreviated schemes of analysis for the use of those who know what they are doing, and the limits of accuracy which will suit their special requirements, are only a few of the points dealt with.

As one example of the interest and value of the bibliographical portion of the work, the "opening up" of chromite for analysis may be selected. It is in the form of footnotes, equal to over a page of ordinary print, and is not merely a bibliography, but has occasional added notes, showing what the references are about and how they may be used. The action of sodium peroxide on various metals used for crucibles is dealt with in such a note, briefly but with surprising accuracy and completeness.

As one of the duties of a reviewer is to act as a critic, it may be justifiable to-point out here that, although the index is an exceedingly good one, it might well be enlarged. So many matters which are of value on general lines, but which can only be spotted through an index, are discussed throughout the book that its value will be much enhanced if the next edition can have a subject-index of sufficient scope to serve as a guide as to what to look for, in addition to a reference index showing where to find what one knows is likely to be there. The important footnote referring to the use of sodium peroxide on p. 475 may be taken as an instance. It does not appear in the index under "peroxide" or "sodium" or "fusion," and is apparently not indexed except under "crucibles"; but the few remarks which are made as to the action of sodium peroxide on different metals appealed particularly to the writer, who has had extensive and expensive experience in experimenting with each kind of crucible mentioned, from the silver vessels used in the days when peroxide was first employed, to those of copper, which, as Dr. Mellor says, are of particular value in many special cases. GEORGE T. HOLLOWAY.

ELEMENTS OF WATER BACTERIOLOGY. By S. C. PRESCOTT and C. A. WINSLOW. New York: John Wiley and Sons. (London: Chapman and Hall.) 1913 Third edition. Price 7s. 6d.

This new edition of a well-known work points not only to the rapid changes which have taken place in our knowledge in regard to this somewhat contentious subject, but also to the appreciation in which the work of these authors is rightly held. In the overwhelming output of scientific papers to-day, the guiding hand of the master is of the greatest value to those who, whilst desiring to keep in touch with progress, have not the time to weigh and test the mass of new facts which are always appearing.

While written from the standpoint of American practice, the deviation from the methods of this country is so little that the work is of as great value to English workers as to those for whom it was primarily written. Further, it shows no signs of "insularity," as the literature of the world is freely quoted, a remark which cannot be made of all American scientific literature.

The various pros and cons of the different bacteriological tests are carefully and impartially discussed, always with the view of showing the direction in which real information is to be obtained, and particularly is this noticeable in the chapters on the isolation of the typhoid organism and on the significance of *B. coli*. In this last, due weight has been given to all, even to the German school, whose views, to many, might seem somewhat reactionary.

Chapter II. on "the Quantitative Bacteriological Examination" is recommended (by the reviewer) as well worthy of study by those who undertake the bacteriological analysis of water in a light-hearted manner, without considering the many pitfalls which beset the way and the absolute necessity for personal attention in the preparation of media if the results obtained are to be of value to the actual investigator, or to be comparable with those of others.

The significance of the different members of the *coli* group and also of the *streptococci* are equally well dealt with, and a chapter on the examination of shellfish has been added, embodying the standard American practice.

The work is to be strongly recommended as a thoroughly reliable guide to all those who are interested in the bacteriological examination of water-supplies, and may also serve the additional good purposes of converting those who still think that such examinations are of less value than chemical analysis, and of substituting a proper and standard technique for methods which have brought, and could only bring into disrepute this most valuable method of controlling the purity of water.

CECIL REVIS.

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INSTITUTE OF CHEMISTRY.

PASS LIST: JANUARY EXAMINATIONS, 1914.

OF eighteen candidates who presented themselves for the Intermediate Examination, seven passed: H. G. T. Boorman, G. A. Bracewell, R. O. Eames, B.Sc. (Wales), J. W. Harris, B.Sc. (Lond.), D. G. Hoyle, R. G. Thin, and J. Young. For the Examination in General Chemistry for the Associateship, one candidate, E. Spencer, A.R.S.M., passed. Of twenty candidates who presented themselves for the Final Examination for the Associateship, ten passed: In the Branch of Mineral Chemistry —E. D. Goddard, B.Sc. (Lond.), F. C. Guthrie, B.A. (Cantab.), and G. S. Heaven, B.Sc. (Lond.). In the Branch of Physical Chemistry—C. W. James, B.Sc. (Wales). In the Branch of Organic Chemistry—R. S. G. Knight, A.R.C.S., B.Sc. (Lond.), S. I. Levy, B.A. (Cantab.), B.Sc. (Lond.), and E. Williams, B.Sc. (Wales). In the Branch of Chemistry of Food and Drugs, Fertilisers and Feeding Stuffs, Soils and Water—B. G. Fagan, B.A., B.Sc. (N.U.I.), A.R.C.S.I., W. Honneyman, and Miss E. G. Mocatta, B.Sc. (Lond.).

APRIL, 1914. Vol. XXXIX., No. 457.

THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

An ordinary meeting of the Society was held on Wednesday evening, March 4, in the Chemical Society's Rooms, Burlington House. The President, Mr. A. Chaston Chapman, F.I.C., occupied the chair.

The minutes of the previous ordinary meeting were read and confirmed.

Certificates of proposal for election to membership in favour of Messrs. R. Bickerstaffe, T. H. Byrom, F.I.C., S. G. Clifford, D. R. Frazer, A.I.C., and J. McLaren, were read for the second time; and certificates in favour of Messrs. Laughlin Dyke Acland, B.A. (Oxon.), 55, Comeragh Road, West Kensington, W., assistant to Mr. E. R. Bolton; Walter Alan Gibbings, P.O. Box Sannomiya 174, Kobe, Japan, factory manager and chemist; Frederic Herbert Lees, F.I.C., 31, Summerhill Road, Dartford, Kent, chief Analyst to Messrs. Burroughs Wellcome and Co., Ltd.; and William Henry Woodcock, 132, Felbrigg Road, Goodmayes, Essex, chief assistant to Mr. Bertram Blount, were read for the first time.

Messrs. R. H. Ellis, A.I.C., and A. de Waele, were elected members of the Society.

The following papers were read: "The Composition and Analysis of Compound Liquorice Powder," by Albert E. Parkes, F.I.C., and Frederick Major, B.Sc.; "A Suggested Simple Method for the Determination of 'Stump' (Wood) Turpentine in American Gum Turpentine," by L. Myddelton Nash, F.I.C.; "The Determination of Carbon Monoxide in Air," by Frank Sturdy Sinnatt and Barnett Joseph Cramer, M.Sc.; "The Composition of the Saline Matter adhering to Certain Wet Salted Skins," by M. C. Lamb; and "Dried Carica Papaya Juice," by F. F. Shelley, F.I.C.

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THE COMPOSITION AND ANALYSIS OF COMPOUND LIQUORICE POWDER.

By A. E. PARKES, F.I.C., and F. MAJOR, B.Sc.

(*Read at the Meeting, March* 4, 1914.)

The analysis of compound liquorice powder, which is a mixture of senna, liquorice root, fennel fruit, sublimed sulphur, and refined sugar, offers some difficulty to the analyst.

No method of analysis or analytical standards are recommended by the Pharmacopœia; and although several analysts have endeavoured to remedy these omissions (Liverseege, *Year-Book of Pharmacy*, 1906, 269; Scott-Smith and Evans, Analyst, 1911, **36**, 198), the only suggested standards at present available are those for moisture, ash, and extract (Gadd, *Year-Book of Pharmacy*, 1901, 390; Moor, "Suggested Standards for Foods and Drugs").

A large number of samples of compound liquorice powder have been examined by us, and our method of examination has been devised with the object, as far as possible, of endeavouring to ascertain the quality of each ingredient, and also with a view to detecting the presence of exhausted drugs.

Samples of senna, liquorice root, and fennel fruit, of undoubted purity were procured, and from these powders were made up and examined similarly, the figures obtained being compared with those calculated from the several ingredients after they had been individually submitted to the same process.

This method, of which the following is a description, can be advantageously used where the amount of sample available is limited in quantity.

Chemical Examination.—Moisture and ash are estimated by the usual methods on the same portion (1 grm.). The soluble ash and alkalinity of same may also be estimated; but the figures for these are of little value, owing to the disturbing influence of the sulphur (Evans, *Pharm. J.*, 1905, **74**, 363).

The carbon disulphide extract is obtained by exhausting 5 grms. with dry carbon disulphide for twelve hours in a Soxhlet. The sulphur in this extract is estimated by oxidising it with fuming nitric acid, and precipitating the sulphuric acid with barium chloride solution in the usual manner. The difference between the sulphur and the total extract is principally oil from the fennel, the senna and liquorice only yielding a comparatively small quantity of extract to carbon disulphide.

The extracted powder, after drying, is mixed with water and made up to a volume of 250 c.c., and allowed to digest, with occasional shaking, for twelve hours. It is then left to settle and the supernatant liquid filtered. The aqueous extract is obtained by evaporating 50 c.c. of this filtrate, representing 1 grm. of powder, to dryness.

Sugar is estimated by treating another 50 c.c. with lead acetate solution, and, after removal of the lead precipitate and excess of lead, polarising, or titrating with Fehling's solution after inversion. The results are usually about 1 per cent. too low. The aqueous extract less sugar gives the extract from the combined drugs.

A further 50 or 100 c.c. is submitted to what, for want of a better name, we have called the "glycyrrhizin test." The solution is acidified with a few drops of dilute sulphuric acid, which precipitates glycyrrhizic acid and traces of other substances. When the precipitate has settled, the liquid is passed through a small filter, and the precipitate washed with a small quantity of cold water. The precipitate is then dissolved in dilute ammonia, passed through the same filter, which is afterward washed with a further quantity of the ammonia solution, the solution being then evaporated to dryness and the crude glycyrrhizin weighed.* Owing to the presence in the aqueous extract of small quantities of other closely allied bodies, it has been found impossible so far to isolate the glycyrrhizin in a pure state.

At present no simple direct method suggests itself for the valuation of the senna, but, providing the carbon disulphide extract and the glycyrrhizin test are satisfactory, a low aqueous extract would point to the probability of the presence of exhausted senna.

The presence of senna is indicated in the glycyrrhizin test. The sulphuric acid precipitates a small amount of material from the senna extract, which on being dissolved in the ammonia solution produces a fine cherry-red colour, easily observed even in the presence of the other colouring matters. This cherry-red colour is probably caused by those closely allied substances—chrysophanic acid and emodin—present in the senna (*cf.* Tutin, *J. Chem. Soc.*, 1913, **103**, 2006). It is not produced in the absence of senna, and we hope to investigate this reaction further to ascertain if it cannot be made available for the estimation of senna extract.

For the estimation of the total sulphur, Squire ("Companion to *British Pharmacopœia*") and Liverseege (*ibid.*) recommend extraction with carbon disulphide. The figure so obtained is, however, at the best only approximately correct, though the loss in sulphur may be balanced by the fennel extract. Sublimed sulphur always contains a quantity of the insoluble variety, and we have found this in some commercial samples to amount to as much as 20 per cent. For the estimation, about 1 grm. of the powder is oxidised with fuming nitric acid and a little potassium nitrate or chlorate, as recommended by Alcock (*Pharm. J.*, 1906, **77**, 485) and Evans (*ibid.*), and the sulphuric acid formed is precipitated with barium chloride. We have found this method to be successful in every case, and fairly rapid. The results obtained are generally about 0·1 per cent. too high.

Substituted drugs must be sought for by the microscope.

Microscopical Examination.—It is essential that the powder should be subjected to a thorough microscopical examination. *Cf.* Scott-Smith and Evans (*ibid.*). The papers on senna by Greenish (*Pharm. J.*, 1901, **66**, 397) and Sayre (*Am. J. Pharm.*, 1896, **68**, 585-592; 1897, **69**, 298-306) are also worthy of note. When examined in 50 per cent. chloral hydrate solution, and glycerol containing a little iodine, the various structures are easily identified—the parenchymatous tissue and characteristic starch grains of the liquorice root, the typical one-celled warty hairs and stomata of the senna, and the oil cells and aleurone grains of the fennel fruit. With little experience it is possible to identify all the different structures; at the same time it is advisable to compare the powder with one made up with ingredients of known purity.

The following list contains the maximum, minimum, and mean figures obtained on thirteen normal samples recently examined. The majority were purchased under the Sale of Food and Drugs Acts, and may be considered typical commercial samples.

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* This gives an indication of the quality of the liquorice root used.

The list also contains the figures obtained on the two prepared samples, and the calculated figures.

TABLE I.

	Maximum.	Minimum.	Mean.	Standard A.	Standard B.	Calculated.
	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.
Loss at 100° C.	7·9	3·0	4·7	3·3	3·9	3·8
Ash	6·2	4·6	5·3	5·2	4·9	4·3
Carbon disulphide extract	10·7	9·2	10·3	9·7	9·5	9·7
Sulphur in extract ...	7·9	6·3	7·1	7·0	6·9	7·1
Extract less sulphur ...	3·6	2·6	3·1	2·7	2·6	2·6
Total sulphur	9·2	7·8	8·6	8·5	8·6	8·4
Cold water extract ...	63·9	61·1	62·6	62·6	62·4	62·4
Ash of extract	2·4	1·8	2·0	1·9	1·9	2·0
Cane-sugar	50·3	48·3	48·8	49·1	48·9	50·7
Extract less sugar ...	14·6	11·8	13·5	13·5	13·5	11·7
Crude glycyrrhizin ...	2·9	1·2	1·9	2·7	2·6	2·0

So far we have not met with any case of substituted or exhausted drugs. Many cases of excess of sulphur and deficiency of sugar have been found which may have been due to careless mixing. For instance, we have found sulphur as high as 20 per cent., and sugar as low as 35 per cent.

The drugs employed in making up the powders referred to above gave the following figures on examination:

TABLE II.

	A.			B.		
	Senna, Tinnivelly.	Fennel, Indian.	Liquorice Root, dec. 2.	Senna Alexandrian.	Fennel, Opt.	Liquorice Root Opt.
	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.
Loss at 100° C.	8·2	9·1	8·9	9·1	10·5	10·1
Ash, total	11·8	13·7	7·0	16·6	11·6	4·1
Insoluble ash	8·8	8·3	5·2	13·3	7·4	1·9
Alkalinity of ash * ...	3·0 c.c.	3·3 c.c.	1·6 c.c.	2·6 c.c.	3·5 c.c.	1·7 c.c.
Carbon disulphide extract	6·4	16·1	1·3	2·9	20·0	0·8
Cold water extract ...	30·5	27·2	40·3	33·7	18·5	29·4
Ash of extract	5·9	7·3	2·5	5·8	4·3	2·4
Crude glycyrrhizin † ...	(3·6)	(0·5)	7·2	(1·7)	(1·8)	7·3
Sugars	1·1	1·8	3·3	1·0	1·8	2·2
Sulphur	0·08	0·37	0·04	0·28	0·41	0·33
Nitrogen	2·34	2·32	1·75	2·31	2·81	1·54
P_2O_5	0·22	1·21	0·09	0·42	1·12	0·22

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* The amount of acid required to neutralise the ash from 1 grm. of material, expressed in c.c. of $\frac{N}{10}$ solution.

† The figures in parentheses represent the percentage of solids obtained by the glycyrrhizin test.

DISCUSSION.

Mr. E. J. PARRY remarked that the proportion of glycyrrhizin in different kinds of liquorice—or at any rate in the extract therefrom, commonly known as liquorice juice—was very variable. For example, the extract from the Spanish root contained about 5 to 7 per cent. of glycyrrhizin; that from Sicilian root about 9 to 13 per cent.; while the least valuable kind—namely, Smyrna root—yielded an extract containing 18 to 22 per cent. of glycyrrhizin. It would therefore be seen that the proportion of glycyrrhizin was by no means an index of the quantity of liquorice present. Moreover, the authors gave results obtained by the glycyrrhizin test in the case of the fennel and senna, but these substances contained no glycyrrhizin at all.

Mr. PARKES said that he quite agreed with Mr. Parry as to the variation in the percentage of glycyrrhizin in liquorice root; but in the compound powders that they had examined the figures were smaller and the variations not so great. The average figure was 1·9, and those given by the standard samples 2·6 and 2·7. According to their calculations, it should be about 2, including the precipitate yielded by the senna and the fennel. They called this "crude glycyrrhizin," it being of course obvious that it was not pure. It was the liquorice, however, that made the chief difference, since the quantities of fennel and senna were comparatively small, and the figures yielded by these ingredients were themselves also small.

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THE DETERMINATION OF CARBON MONOXIDE IN AIR.

BY FRANK STURDY SINNATT AND BARNETT JOSEPH CRAMER,
M.SC. (TECH.).

(*Read at the Meeting, March* 4, 1914.)

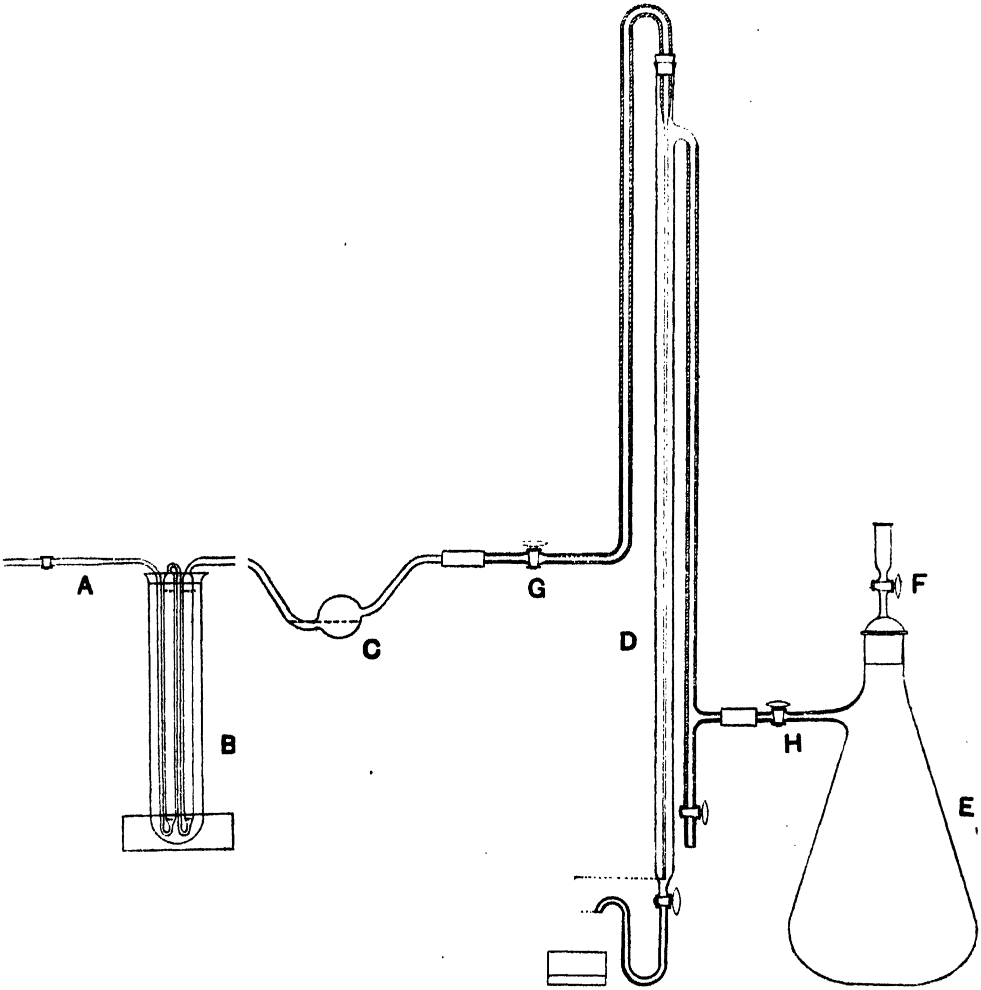
THE determination of small percentages of carbon monoxide in the air of rooms and in other gases is of interest from a technical point of view.

There is a growing tendency on the part of the gas industry to increase the percentage of carbon monoxide in illuminating gas; producer gas is also being applied for heating purposes where formerly coal gas was used. As there appears to be no legal limit to the percentage of carbon monoxide which may be present in commercial gas, the risks of gas-poisoning from escapes and from incomplete combustion of the gases in faulty burners are very much greater, especially when gases are under pressure.

The dangers of poisoning from carbon monoxide are well known, but few statements are available describing the influence of small percentages upon the general health of the workpeople who may be continually exposed to it. Hempel states that decided symptoms of gas-poisoning are observed when air contains 0·05 per cent. of carbon monoxide. It is not, however, possible to determine this percentage by means of the ordinary gas-apparatus.

The method described in this paper has been used for the estimation of carbon monoxide in the waste gases from gas-heated irons and the exhaust gases from a gas-engine.

An examination of the literature of the subject shows that little information of an accurate kind can be found as to the percentage of carbon monoxide in the gases issuing from gas-fires, gas-heated stoves, gas-irons, and exhaust gases, or in the air of rooms in which coal gas or producer gas is being burned. We believe a possible explanation of this may be found in the difficulties encountered in the estimation of the gas.



The most convenient reagent to use for the oxidation of carbon monoxide is iodine pentoxide, as described by Ditte (*Bull. Soc. Chim.*, 1870, **13**, 318), which reacts with the gas according to the following equation:

$$5CO + I_2O_5 = 5CO_2 + 2I.$$

This reaction is quantitative at about 70° C., and the most suitable temperature appears to be 140° to 160° C. As will be seen, the work in this paper confirms this view, since the whole of the analyses have been doubly checked against a second oxidising agent—viz., copper oxide at a red heat. It is necessary to eliminate sulphur dioxide, sulphuretted hydrogen, and unsaturated hydrocarbons, as these also react with iodine pentoxide; but Fillunger (*Chem. Zeit.*, 1903, **27**, 27) and Kinnicutt and Sanford (*J. Amer. Chem. Soc.*, 1900, **22**, 14; ANALYST, 1900, **25**, 160) have shown that iodine pentoxide is not affected by saturated hydrocarbons.

There are practical difficulties in the utilisation of the above reaction. The estimation of either the iodine liberated or the carbon dioxide evolved would serve as a measure of the carbon monoxide present in the gas. The estimation of the iodine has been carried out in various ways, but perhaps the most convenient is the method described by Kinnicutt and Sanford (*loc. cit.*), in which the iodine is collected in a solution of potassium iodide and then titrated with a dilute solution of sodium thiosulphate. These authors also stated that the method which depends upon the absorption of the carbon dioxide produced was unsatisfactory. The iodine pentoxide has to be heated, and pure air passed through it until the iodine has been volatilised and obtained in a form suitable for estimation. This requires considerable time and causes inaccuracies. A single determination may take five to seven hours. Morgan and McWorther (*J. Amer. Chem. Soc.*, 1907, **29**, 1589) confirmed the statements of Kinnicutt and Sanford, but removed the iodine from the gas and then absorbed the carbon dioxide evolved in barium hydroxide solution contained in a long washing-tube. The excess of barium hydroxide was determined by titration with oxalic acid. The same objections may be advanced against this method—viz., the time required in order to obtain perfect absorption of the carbon dioxide by the barium hydroxide solution, and the uncertainty arising from the action of dilute solutions of barium hydroxide on the glass during a long period; the prevention of contact with the air and the breath of the experimenter; and the difficulty of titrating the reaction liquid at the completion of an estimation. That this last objection is recognised is shown by the fact that McFarlane and Gregory (*Chem. News*, 1906, **94**, 133; ANALYST, 1906, **31**, 369) describe a special washing-apparatus which admits of the solution of barium hydroxide being exposed to the gas, and also allows of the actual titration being carried out without the liquid being transferred to a second vessel previous to being titrated.

The method we have devised depends upon the collection of the whole of the carbon dioxide after the carbon monoxide has been oxidised with iodine pentoxide. The gas is obtained in a dry glass flask which admits of the carbon dioxide present being determined by Pettenköfer's process. It is believed that the accuracy of this process will be attainable, which, according to Letts and Blake (*Chem. Soc. Proc.*, 1896, **12**, 192), is 0·04 *parts in* 10,000.

The gas used in the experiments was prepared in the following manner: a measured volume of carbon monoxide (99·8 per cent. carbon monoxide by analysis) was mixed with air which had been freed from carbon dioxide. The mixture was then stored in a gas-holder. The volume of the mixture prepared was about 80 litres. The whole of one set of analyses was carried out with this sample of gas. The gas-holder was of the gasometer type, and the liquid in it remained unchanged. The gas was allowed to stand at least twenty-four hours before an experiment was commenced so as to insure the liquid in the gas-holder being saturated with the gas. The gas was passed through the following reagents contained in washing-bottles: fuming sulphuric acid; aqueous caustic potash; concentrated sulphuric acid; two long tubes containing solid caustic potash, in order to eliminate any carbon dioxide, unsaturated hydrocarbons, and moisture.

When carbon monoxide is estimated by determining the iodine liberated from iodine pentoxide, it is necessary to remove completely the moisture from the gas, since water (or water vapour) decomposes iodine pentoxide with the liberation of iodine. In consequence, special precautions must be taken to dry the gas. Where the carbon monoxide is being measured by the volume of carbon dioxide produced, the presence of moisture does not interfere with the accuracy of the process.

The gas was then passed into the tube *A* on the diagram (see p. 164). The main features of the apparatus can be gathered from the drawing, but since the various parts differ considerably from any previously described for the estimation of carbon monoxide, the following brief description is given:

The leading tube *A* was sealed to the wider tube, filled with alternate layers of iodine pentoxide and glass-wool throughout its length. As will be seen, the gas passes upwards through the iodine pentoxide in both the tubes. The tube is placed in glass vessel *B*, containing paraffin-wax, which was heated on a small sandbath. It was found that the bath could be kept constant within about 3° C. by regulating the height of the flame. This tube, *A*, was sealed to a small washing-tube or catch-bottle, *C*, containing mercury, through which the gas had to travel. This eliminated the iodine from the gas previous to it entering the apparatus *D*. Former experimenters have used metallic aluminium, copper or silver, but mercury was found to be more convenient and efficient. The apparatus *D* was described by one of us (ANALYST, 1912, **37**, 12) as a means for filling an evacuated vessel with gas at any desired rate. It was shown there that the vessel *E* could be made to act as an aspirator, and at the same time, by reading the level of the mercury in *D*, before and after the experiment, supplied the volume of gas which actually passed into *E*. As *D* contains mercury, and *E* is dried previous to an experiment, no loss of carbon dioxide can be possible.

The gas-vessel *E* was specially devised to admit of easy cleaning and drying, and rapid introduction of the barium hydroxide solution; the bore of the tap was made large enough to allow the end of a burette, which had been drawn out, to be passed through it. The barium hydroxide solution could be titrated without opening the flask, the liquid being titrated through the bore of the tap *F*.

A determination was carried out in the following manner: The capacity of the gas-vessel *E* and of the apparatus *D* was ascertained. The vessel *E* was dried and connected to *D*. The two vessels were then swept out with air from which the carbon dioxide had been removed. The taps *F* and *G* were then closed, and the apparatus and flask evacuated by means of a water-pump. Mercury was allowed to enter the main tube of *D*, and the height of the mercury column was read off accurately. The volume of gas-space available in the gas-flask could now be calculated. The iodine pentoxide tube is heated to the required temperature and connected to the train of washing bottles (not shown on the diagram). The gas to be analysed was aspirated through the iodine pentoxide tube and mercury washing-bottle until both were filled with either the gas to be analysed or the oxidation products. This is only necessary at the beginning of a set of determinations. The evacuated flask and apparatus were then connected to the mercury washing-vessel, *C*. Mercury was permitted to flow from the tube of *D* at a suitable rate. As shown by one of us (*loc. cit.*, and *Gas World*, 1913, **58**, 218), the flow of gas through the train of apparatus into the iodine pentoxide tube can be set at any rate by the speed at which the mercury is allowed to pass from the tube. About 1 litre per hour is an average rate. A glance at the results of the experiments will show that various rates of flow have been used. The experiment may be left without supervision until the whole of the mercury has run from the burette. It was found to be more convenient to stop the experiment and read the level of the mercury a second time after adjusting the pressure for the liquid in the washing-bottles. By this means the volume of gas which has passed into the gas-vessel may be calculated, and a certain diminished pressure still remains in the gas-vessel. The tap *H* of the gas-flask is now closed, and the flask disconnected from *D*. A measured excess of standard barium hydroxide solution is run into the small cup on the flask, and allowed to enter the flask as rapidly as possible. No gas is displaced; this eliminates one of the sources of error in the determination of the carbon dioxide in the gas in the gas-flask. The pressure in the flask is brought to that of the atmosphere by allowing air free from carbon dioxide to enter. After being allowed to stand, with occasional agitation, the excess of barium hydroxide is determined by titration with $\frac{N}{20}$ hydrochloric acid. The volume of carbon dioxide found in the gas-flask is a direct measure of the carbon monoxide in the same volume of the gas.

One of us has shown already (ANALYST, 1913, **38**) that this principle can be used for the estimation of carbon dioxide in carbonates, and the extension of the process would point to the method being of use for the estimation of even smaller amounts of carbonates than were attempted in that paper.

Since the gas analysed in these experiments was known to contain only carbon monoxide—no other gas which was oxidisable could be present—it was thought that it would be of interest to repeat the experiments with a second oxidising agent, such as copper oxide. The series of figures show that the oxidation with iodine pentoxide under the conditions described in this paper is as complete as when red-hot copper oxide is used. Further, for many technical purposes copper oxide will find applications where it would be inconvenient to use iodine pentoxide.

The copper oxide was placed in a combustion-tube, and the length of the layer used was about 9 inches. Both Jena glass and silica tubes were tried. The tube was heated in a small furnace to a red heat (750° C.). The tube was connected to the train of washing-bottles, and then to the apparatus *D*. The determination was carried out in exactly the same manner as when using iodine pentoxide. It was found that for gas containing 2 per cent. of carbon monoxide the oxidation of 2 litres could be completed in about thirty minutes. The carbon monoxide present in each sample of gas experimented upon was determined where possible by combustion over red-hot copper oxide, and absorption of the carbon dioxide in weighed potash bulbs. Below a certain dilution this method is impracticable. The method described by Kinnicutt and Sandford (*loc. cit.*) was also used. In all, about one hundred samples of gas were analysed, of which the typical concentrations are given below :

Gas.	Potash Bulbs : Oxidising Agent Hot Copper Oxide.			Absorbing the CO_2 formed in Barium Hydroxide, followed by titration with Hydrochloric Acid.					
				Oxidising Agent Iodine Pentoxide.			Oxidising Agent Copper Oxide.		
		Vol. Gas, C.C.	CO per Cent.	Time, Hours.	Vol. Gas, C.C.	CO per Cent.	Time, Hours.	Vol. Gas, C.C.	CO per Cent.
A.		1882	2·20	3	1959	2·07	3	1959	2·07
		941	2·03	3	2050	1·91	2¼	1779	2·08
		941	2·08	4	1779	2·08	35 mins.	2210	2·17
				2½	1668	2·15	28 ,,	2050	1·90
B.		1902	0·709						
		2872	0·670						
	Carbon Monoxide measured by Iodine liberated from Iodine Pentoxide.								
	Time, Hours.	Vol. Gas, C.C.	CO per Cent.						
	4½	548·0	0·673	5	2268	0·700	1¾	2240	0·682
	6	1017·0	0·679	5	2062	0·650	2	2085	0·676
	5	507·5	0·652	6	2202	0·689	—	—	—
C.	5	2373·0	0·039	3	2228	0·040	2¼	2144	0·042
	3¾	2467·0	0·039	4	2201	0·042	3	2227	0·040
	4	2455·0	0·041	4	2253	0·040	1¾	2226	0·041
	5	2431·0	0·041	—	—	—	—	—	—
D.	4½	2425·0	0·025	4	2182	0·025	4¼	2201	0·024
	5	2320·0	0·025	3¼	2163	0·026	4	2213	0·025
	6½	2436·0	0·023	—	—	—	—	—	—

A number of experiments were carried out on gas A, taking a shorter time than thirty minutes, but the results were all low. It was not possible to check the percentage of carbon monoxide in gas C by means of potash bulbs, the amount present being too small to yield accurate results.

We wish to emphasise certain features of the method described above. Although the apparatus may appear somewhat complicated, it is actually simpler than that necessary for the aspiration and measurement of a large volume of gas with any degree of accuracy. The actual measurement of the volume of the gas is not interfered with by any of the constituents being soluble in the containing liquid. The method is extremely easy to carry out, and a number of workers have tried the method and found no difficulty in the manipulation. The experiments require little attention during the actual passage of the gas; this will be recognised as of importance when the time necessary for carbon monoxide and iodine pentoxide to react quantitatively is considered. When one experiment is complete, another may be commenced at once by simply attaching a fresh gas-flask which has been evacuated in the meantime. Any desired volume of gas may be taken in the same flask, all that is necessary is to leave the contents under a lower pressure.

The method is being applied by one of us to the determination of small percentages of methane in air.

School of Technology,
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Discussion.

Mr. A. E. **Parkes** asked whether the authors had examined any samples of the air of rooms heated by gas-fires, and whether they were able to suggest what proportion of CO in the air of rooms so heated might be regarded as dangerous.

Dr. **Lessing** said the authors had mentioned that reliable figures were not available for the CO contents of air and flue-gases, but during recent years, in connection principally with the testing of gas-fires, some very reliable investigations had been carried out on that subject, involving accurate determinations of CO. For the determination of methane, and also of hydrogen, the copper oxide method had been used with considerable success during the last three or four years, though whether it would yield satisfactory results in the presence of CO had not, he believed, been established. With mixtures from which CO and heavy hydrocarbons had been removed, the copper oxide method could be so finely adjusted that at a comparatively low temperature—*e.g.*, 250° C.—hydrogen only, and no methane, was oxidised, while at 400° to 500° C. the methane would be burnt up. The copper oxide method was, however, principally used when the quantities to be determined were larger than those contemplated by the present authors—as, for instance, in the case of coal-gas.

Mr. G. N. **Huntly** said that some years ago the late Dr. Wade had carried out, for the Local Government Board, an investigation on the products of the combustion of coal-gas, though whether the results had ever been published he did not know. He had assisted Dr. Wade in that work by making the determinations of carbon monoxide, using the iodine pentoxide method. He had preferred to determine the iodine rather than the carbon dioxide, and had found it possible, when certain sources of error were eliminated, to determine accurately a much smaller quantity of iodine than of carbon dioxide, although the former perhaps required more precautions. The iodine pentoxide tube was specially prepared by passing through it air which had been purified by being passed first through another iodine pentoxide tube and then through sulphuric acid, potash, and sulphuric acid again. For the estimation of the iodine a strong solution of potassium iodide was used, and if appropriate precautions were taken, perfectly satisfactory results could be obtained by titration with $\frac{N}{1000}$ thiosulphate solution, it being possible by this method to determine accurately as little as 2 or 3 parts per 1,000,000 of CO in air. In Dr. Wade's investigation the carbon dioxide also had been determined by Pettenköfer's method, and the two methods agreed closely. The method was a selective one, marsh-gas and hydrogen not being attacked. It was only necessary to remove any unsaturated hydrocarbons. As to the temperature of oxidation, 100° C. was quite sufficient, temperatures like 150° C. being not only unnecessary, but likely to introduce errors. In a slow current of gas the CO was completely oxidised at the temperature of boiling water, but it was not advisable to put the tube actually into boiling water, since after forty or fifty hours it would probably crack. It was better to use an alcohol or some other liquid which would not attack the glass.

The authors write in reply that they intend to examine the waste gas from a number of sources. They are in agreement with Mr. Huntly that oxidation is complete at a lower temperature than 160° C., but iodine pentoxide acts more quickly at the higher temperatures. With regard to the introduction of errors, any decomposition of the iodine pentoxide does not influence the result of the estimation as it will when the method in which the iodine liberated is determined.

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STANDARDISATION OF DRIED CARICA PAPAYA JUICE.

By F. F. SHELLEY, F.I.C.

(*Read at the Meeting, March* 4, 1914.)

From time to time tests for *Carica Papaya* juice have appeared in the Analyst, none of which, I think, are so simple as the determination of the amino acids formed by the hydrolysis of a uniform body such as casein. The method I suggest is a modification of Sörensen's test, and is as follows: Four grms. of casein (Hammerstein) are dissolved in 100 c.c. of an alkaline solution containing 4 c.c. $\frac{N}{1}$ soda. To 25 c.c. of this solution are added 25 c.c. of water containing 0·1 grm. of dried *Papaya* juice, and the mixture digested in an incubator at 37° C. for four hours. To 20 c.c. of the liquid are added 10 c.c. of a 40 per cent. solution of formaldehyde just neutralised with $\frac{N}{5}$ soda. The mixture, equivalent to 0·04 grm. of dried *Carica Papaya* juice, is titrated with $\frac{N}{5}$ soda, and at least 1 c.c. should be required to neutralise the amino acids formed (using phenolphthaleïn as an indicator), after subtracting the c.c.'s required by a similar mixture without dried *Papaya* juice. Pure dried *Carica Papaya* should give no reaction for starch with iodine, nor reduce Fehling's solution before or after hydrolysis. My experiments were carried out on a pure specimen which had been very kindly furnished to me by Dr. Francis Watts, C.M.G., Commissioner of Agriculture for the West Indies.

0·04 grm. papain forms amino acid equivalent to 0·45 c.c. $\frac{N}{5}$ alkali, whilst 0·02 grm. of pancreatin, tested in the same way, but digesting for one hour only, should require at least 2 c.c. $\frac{N}{5}$ soda.

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

Detection of Beef-Fat in Lard. A. Bömer, R. Krönig, R. Ley, and H. Merten. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1914, **27**, 153-172.)—Further investigation has shown that the presence of coconut oil, earthnut oil, sesame oil, and cottonseed oil does not interfere with the results obtained by a method described recently for the detection of beef-fat in lard (ANALYST, 1914, 84); hydrogenised oils (hardened oils), however, behave like beef-fat, and in this case the presence of vegetable oils must be ascertained by means of the phytosterol acetate test. The difference between the melting-point of the insoluble glycerides of lard and that of the fatty acids is not affected by feeding pigs on maize, coconut, or other oil meals. Results of a few experiments are given indicating that the method may be of use in detecting the presence of lard in butter. W. P. S.

Cacao Seeds. L. Reutter. (*Bull. Agric. Intell. and Plant Diseases*, 1913, **4**, 1213.)—A white crystalline substance soluble in water was isolated from cacao seeds which the author terms cacaorina. The substance is obtained by the spontaneous crystallisation of a methyl alcohol extract of the seeds. On hydrolysis it yields theobromine and cacao red; the latter on treatment with dilute sulphuric acid gives rise to a dextro-rotatory sugar and a new substance, cacao brown. H. F. E. H.

Estimation of Water in Cheese by Mai and Rheinberger's Method. Weigmann and E. Haglund. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1914, **27**, 77-83.)—This method (ANALYST, 1913, **38**, 146) was found to yield results varying from 1·84 to 3·74 per cent. higher than those found by the ordinary method of estimating the water by grinding the cheese with sand and drying the mixture at 105° C. until practically constant in weight. The results obtained by the latter method agreed closely with the figures found on subtracting the sum of the percentages of the other constituents from 100 (estimation of water by "difference"). The distillate in Mai and Rheinberger's method contains ammonia when the distillation temperature exceeds 150° C., but this ammonia is not derived solely from products formed during the ripening of the cheese, since freshly prepared curd also yields an ammoniacal distillate. W. P. S.

Method for Calculating the Value of Foods from the Point of View of Milk Production. N. Hansson. (*Bull. Agric. Intell. and Plant Diseases*, 1914, **5**, 84-87.)—The differences observable between Kellner's starch values for various fodders and fodder mixtures and the Swedish and Danish food-unit values depend upon the fact that Kellner's figures are the result of fattening experiments with steers, while the latter are founded on experiments with milch cows. In other words, the starch values measure fat-formation value, while the food-units measure milk-production values. Fats, carbohydrates, and crude fibre, have the same value in the two cases, but protein is more advantageously used in the production of milk proteins than in fat formation. The author has introduced a method for calculating the milk-producing value, which only differs from Kellner's for the calculation of the starch value as regards the factor for the reduction of protein (1·43 in place of Kellner's 0·93). The two values work out very nearly the same for fodders poor in protein, such as hay, green fodder, and cereals. The principle of recognising in foods rich in protein a feeding value higher than their starch value is not opposed to Kellner's opinion on the starch value, as he calculates the pecuniary value of the foods on the basis of a fundamental price, not only per kilo of starch value, but also per kilo of protein. Below are a few of the examples given:

MILK-PRODUCING VALUE OF FOODS.

	Content of Digestible Matter.					Value for the Production.			1 Food-Unit = Kilo of Fodder.	Ratio of Milk-Producing Unit to Food-Unit.
	Protein.	Starch.	Fat.	Nitrogen Free Extract.	Fibre.	Total Relative Value (Wertigkeit), Kellner.	Per 100 Kilos.			
							Starch Value.	Milk-Producing Value.		
Earth-nut Cake ...	39·6	1·4	7·2	21·2	0·7	98	74·9	94·0	0·8	1 : 0·75
Barley ...	6·5	1·0	1·7	67·0	1·4	99	72·0	75·2	1·0	1 : 0·75
Peas... ...	17·2	3·0	0·9	48·8	2·5	98	68·0	76·2	1·0	1 : 0·76
Meadow-hay ...	4·2	1·3	1·0	24·3	15·5	64	29·2	30·5	2·5	1 : 0·67
Swedes ...	0·5	0·5	0·1	8·1	0·8	87	8·3	8·5	9·0	1 : 0·77

H. F. E. H.

Detection of Formaldehyde and of Formaldehyde Compounds by Means of Schiff's Reagent containing Hydrochloric Acid. H. Fincke. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1914, **27**, 246-253.)—The reagent is prepared by dissolving 1 grm. of rosaniline hydrochloride in 500 c.c. of water, adding 25 grms. of crystallised sodium sulphite and 15 c.c. of hydrochloric acid, sp. gr. 1·124, and diluting the mixture with water to 1 litre. Ten c.c. of the liquid to be tested are mixed with 2 c.c. of hydrochloric acid (sp. gr., 1·124), and 1 c.c. of the reagent is added. If formaldehyde is present, a red-violet coloration develops within a period varying from a few minutes to twelve hours. The reaction is characteristic of formaldehyde, other aldehydes not yielding a coloration in the presence of the excess of hydrochloric acid used. The test is best applied directly to the liquid under examination (the latter may be decolourised with animal charcoal, if necessary). Attention is drawn to the fact that certain organic substances combine with small quantities of formaldehyde, and the latter is not liberated even by distillation with a mineral acid. Minced rhubarb leaves decompose or combine with formaldehyde to an appreciable extent, and the same may be said of urea, although in this case the greater part of the formaldehyde is liberated when the solution is acidified. Milk, fruit juices and honey combine with but very small quantities of formaldehyde. As a rule the reagent is capable of detecting the presence of 1 part of formaldehyde in 400,000 parts of liquid. Hexamethylenetetramine also yields a coloration with the reagent, 1 part in 200,000 being detectable, but formaldehyde combined previously with sulphurous acid does not give the reaction. W. P. S.

Estimation of Husks in Cottonseed Cakes and Meals. C. Grimme. (*Chem. Zeit.*, 1914, **38**, 137-139.)—Fraps has proposed a chemical method (*J. Soc. Chem. Ind.*, 1909, **28**, 590) depending on the differential hydrolysis of the hulls and kernels, by boiling dilute sodium hydroxide. Trouble is, however, experienced from frothing, and the filtration of the alkaline liquid is very slow. The author now describes a similar method in which hydrochloric acid is employed instead of the alkali: 2 grms. of the finely ground material, previously freed from fat, are treated in an Erlenmeyer flask with 200 c.c. of 1 per cent. hydrochloric acid. The mixture is boiled under a reflux condenser over a moderate flame for half an hour reckoned from the commencement of boiling. Frothing will give no trouble if the mixture be heated carefully; but if it should occur, any particles of meal carried into the upper part of the flask should be washed down again with 1 per cent. acid. After boiling, the flask is filled with hot water, and the residue is immediately collected on an asbestos filter connected with a pump, and washed with hot water, then with alcohol and ether. The filter and residue are transferred to a platinum dish, dried at 100° C., and weighed; the weight of the residue is then found by difference after incineration. The percentage of husks is calculated by means of a formula empirically established: $x = \frac{(y-11)\,100}{70-11}$, where x is the percentage of husks, and y the percentage of dry residue from the hydrolysis. The values 70 and 11 represent average percentage residues found on treating pure separated husks and kernels respectively by the same process. The values vary very slightly from the average for cottonseed products of diverse origins; only seeds from Togoland show rather lower results for the husks. Test experiments with mixtures showed that the method gives results accurate to ±0·6 per cent.; they agree well with the results obtained by Fraps's method, and fairly well with those found by sifting. J. F. B.

Estimation and Significance of Ammonia in Milk. J. Tillmans, A. Splittgerber, and H. Riffart. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1914, **27**, 59-76.)—The estimation of the quantity of ammonia present in milk is preferably carried out on the serum obtained by treating the milk with mercuric chloride and hydrochloric acid (two volumes of milk with one volume of a mixture of equal parts of 5 per cent. mercuric chloride solution and 2 per cent. hydrochloric acid), or with basic lead acetate and sodium phosphate (400 c.c. of milk are treated with 40 c.c. of basic lead acetate solution, filtered, and 240 c.c. of the filtrate are mixed with 27 c.c. of saturated sodium phosphate solution, and again filtered). The ammonia is then estimated by distilling the serum under reduced pressure in the presence of magnesium oxide, or by treating the lead acetate serum with magnesium chloride and a further quantity of sodium phosphate, and rendering the mixture slightly alkaline with sodium hydroxide solution; the precipitated ammonium magnesium phosphate is then collected and distilled with magnesium oxide under the ordinary atmospheric pressure. Both methods yield trustworthy results. Milk fresh from the cow contains from 3 to 4 mgrms. of ammonia per litre; this quantity increases when the milk undergoes decomposition. The ammonia-content is not altered when the milk is heated, but, owing to the formation of substances containing ammonia which are precipitated by the magnesium chloride method of estimating the ammonia, this method yields higher results in the case of heated milk than does the distillation process. The addition of small amounts of cow-dung to milk does not immediately increase the quantity of ammonia present, but the ammonia increases more rapidly in such milk on keeping than it does in the case of clean milk. Whilst the quantity of ammonia in a milk affords no evidence of the presence of added impure water, large quantities indicate that the bacteria have increased to an undesirable extent. In the authors' opinion a milk containing more than 10 mgrms. of ammonia per litre cannot be considered to be fresh, and should not be used for feeding infants. W. P. S.

Electrical Conductivity of Milk. Electrometric Determination of End-Point in Manufacture of Sweetened Condensed Milk. L. C. Jackson, L. McNab, and A. C. H. Rothera. (*J. Soc. Chem. Ind.*, 1914, **33**, 59-60.)—When separated milk is concentrated, the electrical conductivity increases until the concentration of solids approximates to 28 per cent., beyond which point further concentration causes the conductivity to diminish. Since the rate of change of conductivity in the neighbourhood of the maximum is small, conductivity measurements are of no value in determining the degree of concentration of unsweetened condensed milk.

With sweetened milk, however, the case is different. When whole milk has 16 per cent. of sugar added to it, and is then concentrated, the maximum conductivity is reached when less than 10 per cent. of the water has been evaporated, and further concentration causes the conductivity to diminish at a rate which accelerates rapidly as concentration is pushed to the usual limit. The actual conductivity both of the original and the concentrated milk varies with the season, and, of course, with the temperature of observation. It is proposed therefore to enclose in the vacuum pan a closed resistance cell containing a satisfactory sample of condensed milk from the previous week's operations as well as an open conductivity cell, which is either of the same dimensions as the resistance cell or has been compared with the latter. In the simpler case, where the two cells have the same dimensions, the process is stopped when the two resistances are equal. More often the cells will not be exactly alike, but, the principle of the method being understood, the means to be taken for the proper balancing of the resistances will be obvious. G. C. J.

Detection of Extracted Paprika. G. Heuser and C. Hassler. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1914, **27**, 201-209.)—A direct estimation of the iodine value of paprika affords information as to whether the sample under examination consists of genuine or extracted material. One grm. of the sample is mixed with 20 c.c. of chloroform, Hübl's solution is added, and, after six hours' contact, the mixture is titrated with thiosulphate solution in the usual way. In the case of genuine paprika, the iodine value thus obtained varies from 31·80 to 34·80, whilst samples which have been extracted, or which are deficient in active principles from other causes, yield values varying from 19·42 to 20·74. It is also shown that the ether extract may be estimated rapidly by a method similar to the Gottlieb-Rose process for the estimation of fat in cheese; the results obtained agree within 1 per cent. with those found by the ordinary ether extraction method. W. P. S.

Detection of Saponin. J. Rühle. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1914, **27**, 192-200.)—The dyes known under the names of "raspberry-red," "lemon-yellow," "emerald-green," "strawberry-red," and "May-green," and used for colouring effervescent beverages, do not possess any hæmolytic action, and their presence in a beverage does not therefore interfere with the detection of saponin by the method described previously by the author (ANALYST, 1912, **37**, 350). Two sapogenins obtained from soap-root and one from quillaia bark were found to be from four to ten times more soluble in ether than their corresponding saponin; these three sapogenins exhibited a feeble but distinct hæmolytic action. Two saponins isolated from guaiacum bark (an acid saponin and a neutral saponin) showed hæmolytic activity in dilutions of 1 : 5,000. The author is of opinion that the addition of saponin to aerated beverages should be prohibited. W. P. S.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Gas-Volumetric Estimation of Free and Combined Amino-Nitrogen in Urine. D. D. van Slyke. (*J. Biol. Chem.*, 1913, **16**, 125-134; through *Chem. Zentralbl.*, 1914, I., 684-685.)—The method of estimating the total amino-nitrogen in urine (ANALYST, 1911, **36**, 17, 457) has been simplified by evaporating the alkaline filtrate to dryness instead of expelling the ammonia by boiling. Instead of washing the precipitate, the liquid is made up to a definite volume, and an aliquot portion used for the estimations. Free amino-acids may be estimated alone after destruction of the urea by soya-bean urease, which neither liberates combined amino-nitrogen nor attacks free amino-compounds. Examples are given showing the applicability of the method to the estimation of hippuric acid. C. A. M.

Estimation of Amino-Nitrogen in Organic Tissues. D. D. van Slyke. (*J. Biol. Chem.*, 1913, **16**, 187-195; through *Chem. Zentralbl.*, 1914, I., 685.)—The amino-acids are extracted from the tissues with hot water containing 0·1 per cent. of 50 per cent. acetic acid, the extract is evaporated to a small volume, and any uncoagulated albumin precipitated with absolute alcohol. The filtrate is made alkaline and freed from alcohol and ammonia by concentration *in vacuo*, and the amino-nitrogen estimated in the residue by means of the author's method (see preceding abstract). From the speed with which the amino-acid reacts with the nitrous acid, and from the relatively small increase produced by hydrolysis with hydrochloric acid, it appears that the nitrogen estimated by this method is derived almost exclusively from free amino-acids. Only a small percentage of amino-nitrogen originates from proteins or their derivatives, or from amines not derived from albumin. C. A. M.

Estimation of Lipoids in Blood-Serum. **L. Grimbert and M. Laudat.** (*J. Pharm. Chim.*, 1914, **9**, 97-104; 145-149.)—The following process is described for the estimation of free and combined cholesterol, phosphorus compounds (lecithins), fatty acids, and fat in small quantities of blood. It consists in treating the blood-serum with alcohol to remove proteins, estimating the free cholesterol by precipitation with digitonin, saponification of the lecithins, fats, and cholesterol esters, and estimating these separately, the fatty acids being found by difference.

Twenty c.c. of the serum are mixed with 100 c.c. of 95 per cent. alcohol, and, after the lapse of six hours, the mixture is filtered; the insoluble portion is then extracted with boiling alcohol in a modified Soxhlet apparatus, the extract is mixed with the alcoholic filtrate, the mixture is evaporated, and the residue obtained is dried for one hour at 50° C. The residue is now extracted with anhydrous ether, the ethereal solution evaporated, and the residue obtained dried at 50° C., and weighed. A quantity of 0·2 grm. of this dry ethereal extract is then dissolved in 95 per cent. alcohol, and the cholesterol precipitated with digitonin, as described by Windhaus (ANALYST, 1910, **35**, 256). The alcoholic filtrate and washings from the digitonin precipitate are evaporated to 20 c.c., and 150 c.c. of ether are added; the excess of digitonin is thus precipitated, and, after its removal, the ethereal solution is evaporated, and the residue saponified with alcoholic potassium hydroxide solution. After the removal of the alcohol by evaporation, the solution is diluted with water, acidified with nitric acid, and extracted with ether. The ethereal solution is evaporated, the residue dried at 50° C. and weighed, and the cholesterol in it (cholesterol present in the serum in the form of esters) is estimated as described above. The aqueous acid solution is evaporated, the residue incinerated, and the phosphoric acid is estimated by the molybdate method; the weight of the phosphomolybdate precipitate divided by 2·3 gives the quantity of phosphorus compounds expressed as distearyl-lecithin. The amount of oleic acid combined with the cholesterol is found by multiplying the weight of the latter (*i.e.*, of the combined cholesterol) by 0·73; this quantity, together with that of the combined cholesterol and of the stearic acid combined with the lecithin, is subtracted from the weight of the ether residue in order to obtain the weight of the total fatty acids present (free fatty acids *plus* fatty acids liberated by the saponification from the fats). The following results were obtained with serum from normal blood, the figures expressing grms. per litre: Cholesterol, free, 0·36 to 0·65; cholesterol, combined, 1·38 to 1·95; lecithin, 1·19 to 2·10; total fatty acids, 2·50 to 4·93.

The total cholesterol may be estimated by saponifying the alcoholic extract of the serum, extracting the fatty acids and cholesterol with ether, evaporating the ethereal solution, and weighing the residue. This residue is then dissolved in alcoholic potassium hydroxide solution and the cholesterol is extracted with petroleum spirit and weighed.

An alternative method consists in estimating the cholesterol colorimetrically. Two c.c. of the serum are mixed with 13 c.c. of 0·6 per cent. alcoholic sodium hydroxide solution, and the mixture is extracted with ether; the ethereal extract is washed with water, evaporated, and the residue is dissolved in 5 c.c. of chloroform. This solution is treated with 2 c.c. of acetic anhydride and 3 drops of $\frac{N}{1}$ sulphuric acid, and, after thirty minutes, the green coloration obtained is compared with that produced by a known quantity of cholesterol under similar conditions.

W. P. S.

Determination of Early Stages of Pregnancy. Bernhardt. (*Zeitsch. für Gest. und Pferd.*, 1913, **7**, 145-148; through *Bull. Agric. Intell. and Plant Diseases*, 1913, **14**, 1745-1748.)—Enzymes capable of breaking down placenta-protein are produced in the blood of animals as the result of pregnancy, disappearing again in from ten to fourteen days after the birth of the young. The author describes Abderhalden's method of determining pregnancy in its earliest stages by testing the blood-serum for the presence of such enzymes, a preparation of placenta-protein foreign to the species of animal under investigation being employed for this purpose. Two or 3 c.c. of the animal's blood-serum are placed in a carefully-tested membrane with 1 grm. of well-shredded placenta-protein free from peptone, the whole being placed in a flask containing 20 c.c. of water and a little toluene, and incubated at 37° C. for sixteen hours. At the end of this period 10 c.c. of the dialysed solution and 0·2 c.c. of 1 per cent. ninhydrine (triketo-hydrindene hydrate) are heated together, and if after cooling a deep blue colour is produced, then the blood contains an enzyme capable of breaking down placenta-protein, and the animal is pregnant. Should yellowish-red or brownish tints appear the blood is too acid or too alkaline, and the experiment must be repeated. If the dialysed solution does not react with ninhydrine, the animal is not pregnant.

H. F. E. H.

Comparison of Various Preservatives for Urine. W. M. Dehn and F. A. Hartmann. (*J. Amer. Chem. Soc.*, 1914, **36**, 409-417.)—So complex a solution as urine cannot be preserved unaltered by any one substance; it is therefore necessary to select such an antiseptic as is best suited to the particular ingredient or ingredients it is desired to estimate. Preservatives which interfere with the accuracy of dextrose determinations are chloroform, gallic acid, phenol, salicylic acid, formaldehyde, hydrogen peroxide, and thymol. Since thymol absorbs iodine and reduces alkaline picrate solution, its presence affects not only the dextrose, but also uric acid and creatinine determinations. Salicylic acid is the best preservative of those studied. Chloroform, though it preserves urea and therefore ammonia, is ineffective with dextrose and creatinine. Boric acid is of little use in 0·2 per cent. concentration. Ether in large quantities preserves well, but volumetric relations are disturbed. Formaldehyde is unsatisfactory since it precipitates urea and is a reducing substance. Hydrogen peroxide interferes with too many reactions in urine analysis, and has feeble bactericidal power. Hydrochloric and gallic acids have nothing to recommend them, while sodium benzoate is a very poor, and sodium borate a poor, preservative. Toluene preserves uric acid and creatinine well. Sodium arsenite is a fair preservative of urine, and strychnine sulphate a good one. Since the activity of many bacteria is accompanied by the formation of phenol, it is not to be expected that this substance will be serviceable, and its presence interferes with the analysis of many components of urine. Sandal-wood oil appears to have considerable value.

H. F. E. H.

ORGANIC ANALYSIS.

Estimation of Acetone. W. M. Marriott. (*J. Biol. Chem.*, 1913, **16**, 281; through *Chem. Zentralbl.*, 1914, I., 821.)—In connection with the estimation of butyric acid the author found that Messinger's method for the estimation of acetone (*Ber.*, 1888, **21**, 3336) gave accurate results, even with small quantities. Scott-Wilson's method (*J. of Physiol.*, 1911, **42**, 444), in which the acetone is precipitated as a condensation product with mercuric cyanide, and the mercury estimated by titration with thiocyanate, was accurate in absence of aldehydes, chlorides, hydrogen sulphide, and ammonia. From very dilute solutions the acetone should be collected by distillation in a small quantity of water. O. E. M.

Estimation of Small Quantities of Acetone in the Nephelometer. W. M. Marriott. (*J. Biol. Chem.*, 1913, **16**, 289; through *Chem. Zentralbl.*, 1914, I., 821.)—Very small quantities of acetone, such as occur in blood, are estimated in the Richards nephelometer by comparing the turbidity obtained on distilling the acetone into silver mercury cyanide solution with the precipitate obtained with a standard solution. The reaction is distinct with 0·01 mgrm. of acetone in 50 to 100 c.c. of solvent. O. E. M.

Separation of *d*-Alanine and *d*-Valine. P. A. Levene and D. D. van Slyke. (*J. Biol. Chem.*, 1913, **16**, 103-120; through *Chem. Zentralbl.*, 1914, I., 574-575.)—In 10 per cent. sulphuric acid solution *d*-alanine combines with phosphotungstic acid in the proportion of 1 : 14 by weight, a crystalline salt being formed. At 0° C. alanine dissolves to the extent of only 0·15 grm. in 100 c.c. of a solution containing 10 per cent. of sulphuric acid and 20 per cent. of phosphotungstic acid (in excess of the quantity combining with the alanine). Under the same conditions, the solubility of *d*-valine is 1·21 grm. per 100 c.c. By alternate crystallisation of the valine as free amino-acid and of alanine as phosphotungstate, a practically quantitative separation of the two substances can be effected. W. P. S.

Estimation of Asphaltic Matters in Dark Mineral Oils. D. Holde and G. Meyerheim. (*Chem. Zeit.*, 1914, **38**, 241-244, 264-265.)—The usual methods for the estimation of asphalts in mineral oils consist in precipitating these matters either with a mixture of alcohol and ether, or with petroleum spirit of sp. gr. 0·695-0·705 ("normal benzine"). Alcohol and ether precipitate, together with the hard asphalt, contain quantities of soft, transition, asphaltic-oily products as well as of ceresin, and it is necessary to distribute the precipitate on sand and charcoal and to extract the product with boiling alcohol. Petroleum spirit also in certain cases precipitates small quantities of ceresin together with the hard asphalt, and an extraction is desirable also in this case. With lubricating oils the quantity of asphalt precipitated by "normal benzine" should not exceed 0·5 per cent. Kantorowicz (ANALYST, 1914, 43) refers to the drawbacks of the alcohol-ether method and proposes hot ethyl acetate as an effective precipitating agent in substitution for the former, and to a certain extent for petroleum spirit also. Kantorowicz claims that ethyl acetate is equivalent to the usual reagents for precipitating hard asphalt, and possesses the advantage of holding in solution the oily products and ceresins, thereby avoiding the tedious extraction treatment. The authors show, however, that hot ethyl acetate is only a very imperfect precipitating reagent for the hard asphalts, and yields far smaller quantities of asphalt than the alcohol-ether method combined with extraction. The results obtained by the latter treatment are approximately equivalent to those found by using light petroleum (boiling below 50° C.) or by the use of *n*-pentane, if preference be given to a reagent of uniform composition. "Normal benzine" gives lower results than alcohol-ether or light petroleum, but in many cases hot ethyl acetate gives lower values still and often shows oils to be free from asphalt, or to contain merely traces when the other reagents separate considerable quantities of hard asphalt. "Normal benzine" is probably the most convenient reagent, and has been in general use for a long time; the contention of Kantorowicz (*loc. cit.*) that it contains variable and considerable quantities of cyclic hydrocarbons is proved by the authors to be unfounded, and it appears probable that these hydrocarbons do not exceed 8 per cent. by volume. If it should be found that cyclic compounds are liable to occur and interfere with the standard precipitating properties, it may be necessary to amend the specification of "normal benzine" and provide for a lower specific gravity, 0·685-0·695 instead of 0·695-0·705, and stipulate that on separation into fractions at 5° C. intervals, no fraction should have a higher sp. gr. than 0·700.

J. F. B.

Direct Estimation of Caoutchouc in Crude Rubber. Pontio. (*Ann. Chim. anal.*, 1914, **19**, 60-62.)—This paper is mainly adverse criticism of the method proposed by Marquis and Heim (ANALYST, 1913, **38**, 511). This and all other methods depending on the use of solvents of low boiling-point are condemned on the ground that such solvents do not dissolve rubber completely, unless it be quite freshly coagulated or precipitated. The greater the nerve and the older the preparation, the more does it resist the action of such solvents. If a solvent of higher boiling-point, such as xylene, is employed, the solution filtered, concentrated under reduced pressure, and poured into alcohol, fair results are obtained, results quite as good as those yielded by any direct method yet described. The result should be compared with that obtained by difference. If they agree well, the result obtained indirectly receives useful confirmation; but in case of disagreement the author thinks that, in the present state of our knowledge, the value arrived at indirectly from the results of a careful analysis along the ordinary lines must be preferred to the value obtained by any direct method yet described. He expresses the view that the solution of the problem of estimating rubber directly will finally be found in some development of Budde's tetrabromide method, but no modification of it yet described can be recommended.

G. C. J.

Note on Synthetic Caoutchouc from Isoprene. G. Steimmig. (*Ber.*, 1914, **47**, 350-354.)—Harries concluded that the caoutchouc obtained from isoprene by the action of heat or of glacial acetic acid is identical in constitution with Para caoutchouc. The diozonides are decomposed with the same velocity, yielding the same products

—viz., lævulinic aldehyde and lævulinic acid, corresponding with a hydrocarbon of the constitution 1.5-dimethyl-cyclo-octadiene (1.5). The author has re-examined the caoutchouc obtained by the polymerisation of isoprene by heat, as well as caoutchoucs prepared by other methods of polymerisation, and has found that the decomposition of the ozonides in all cases yields, not only the products mentioned above, but also quantities of succinic acid and acetonyl-acetone which would correspond to the presence of about 20 per cent. of the isomeric hydrocarbon 1.6-dimethyl-cyclo-octadiene (1.5). Natural caoutchouc, on the other hand, differs from all the synthetic caoutchoucs hitherto examined, in that it yields none of the derivatives of the 1.6 hydrocarbon, and appears to be a uniform product containing only the 1.5 ring. Constitutionally the 1.5 hydrocarbon may be formulated by the combination of two isoprene molecules in a symmetrical manner, whilst the 1.6 hydrocarbon results from their combination asymmetrically. J. F. B.

Detection of Carbon Disulphide in Extracted Oils. Utz. (*Farbenzeit.*, 1913, **19**, 698-700; through *Chem. Zentralbl.*, 1914, I., 578.)—The test depends on a reaction between carbon disulphide and thalloacetylacetone (*cf.* Kurowski, *Ber.*, 1910, **43**, 1079), an orange-yellow precipitate being formed. The oil to be tested is distilled after the addition of one-fourth its volume of alcohol, and the distillate is mixed with the reagent; in the presence of carbon disulphide, an orange-yellow precipitate forms at once or after the lapse of a short time. The test will detect 1 per cent. of carbon disulphide in an oil, and the reaction is not given by oils, such as rape oil, which contain sulphur. W. P. S.

Picrate Colorimetric Method for the Estimation of Carbohydrates. W. M. Dehn and F. A. Hartmann. (*J. Amer. Chem. Soc.*, 1914, **36**, 403-409.)—The method depends upon the formation of a red to brown colour when solutions of sugars are heated with sodium carbonate solutions of picric acid. Since the colour formed does not match well with solutions of ferric acetate, potassium dichromate or *o*-nitrophenoxide, the colour of the sugar-picrate solution itself is employed as the standard of colour, any desired sugar being used for this purpose. The picric acid solution contains 2 grms. of the acid and 4 grms. of anhydrous sodium carbonate per litre. An excess of picric acid is necessary to insure complete oxidation and to prevent the formation of colourless reduction products of the picric acid (Chapman, ANALYST, 1909, **34**, 475). The standard colour solution is prepared by inverting 0·95 grm. of cane sugar in about 100 c.c. of water with hydrochloric acid, cooling, adding an excess of sodium carbonate and 100 c.c. of the picrate solution. The whole is then boiled for five minutes after the colour begins to develop, and is then diluted to 1,000 c.c. As this solution is too dark for direct use in the colorimeter, it is diluted ten times (0·01 per cent.). The colour readings may be made in any suitable colorimeter, such as Duboscq's, or bottles of clear glass may be employed. Since the standards are not strictly proof to sunlight, they are best kept in the dark, but their rate of deterioration is slow. Artificial light should not be employed. A maximum error of 1 per cent. may be expected. For the determination of lactose in milk, 10 c.c. of the milk are run into a 250 c.c. flask with 50 to 100 c.c. of water and

1 c.c. of 50 per cent. sulphuric acid. The mixture is heated on the sand-bath until flocks of casein separate or until the solution boils. The whole is then cooled, made up to volume, filtered, and 6·25 c.c. of the filtrate run into a 250 c.c. flask. Two or three volumes of water are then added, an excess of sodium carbonate solution, and 10 c.c. of the picrate solution. The whole is heated on the sand-bath for five minutes after the colour begins to develop, then cooled and diluted to 250 c.c. The colour is matched against a standard solution of lactose. H. F. E. H.

Method of Estimating Colophony in Fats and Soaps. Leiste and C. Stiepel. (*Seifensieder Zeit.*, 1913, **40**, 1233–1234; through *Chem. Zentralbl.*, 1914, I., 577.)—The method depends on the solubility of resin-soap in acetone. Two grms. of the soap are dissolved in 20 c.c. of alcohol, the solution is neutralised with sodium hydroxide, and evaporated to a paste, which is then mixed thoroughly with 10 grms. of sand and dried completely in the water-oven. The dry mass is now extracted eight successive times with aqueous acetone, using 10 c.c. of the latter each time (this aqueous acetone is prepared by the addition of 2 per cent. by volume of water to acetone which has been dried over anhydrous sodium sulphate), the acetone solutions are filtered into a weighed flask, evaporated, and the residue is dried until constant in weight. Should the acetone solution yield a precipitate when evaporated to one-half its volume and then cooled, it must be refiltered. The weight found gives the quantity of resin-soap present. Any unsaponifiable matter contained in the sample will be included in the resin-soap, but it is pointed out that commercial soaps are prepared from fats containing but small quantities of unsaponifiable matters.

W. P. S.

Estimation of Glycerol in Technical Glycerines and Soap Lyes. M. Tortelli and A. Ceccherelli. (*Chem. Zeit.*, 1913, **37**, 1505-1506, 1573-1574; 1914, **38**, 3-5, 28-31, 46-47.)—From a critical examination of the different methods of estimating glycerol, the conclusion is drawn that the only one capable of giving accurate results under all conditions is Hehner's dichromate method, with certain modifications, as described below: *Glycerol in Crude Commercial Glycerines:* About 5 grms. of the sample are mixed with 10 c.c. of water and 5 c.c. of basic lead acetate solution (prepared by boiling 10 per cent. lead acetate solution for five hours with excess of lead oxide), then shaken with 5 c.c. of 10 per cent. sodium sulphate solution, and made up to 100 c.c. and filtered. Ten c.c. of the filtrate are treated with a few drops of dilute sulphuric acid, and then with 50 c.c. of potassium dichromate solution (74·564 grms. per litre) and 50 c.c. of dilute sulphuric acid (1:1), and the whole gently boiled for fifteen minutes over a flame. When completely cold, the excess of dichromate is titrated with ferrous ammonium sulphate solution (200 grms. per litre), with potassium ferrocyanide as indicator. From the results of this preliminary estimation of the glycerol, the amount of the sample to be taken for an accurate estimation is found by reference to the table below. This quantity, which should contain about 0·5 grm. of pure glycerol, is diluted to 250 c.c., and 25 c.c. of the mixture are shaken for five to ten minutes in a glass-stoppered flask with silver carbonate prepared immediately before use by mixing 4·9 c.c. of $\frac{N}{1}$ sodium carbonate solution with 140 c.c. of 0·5 per cent. silver sulphate solution, and washing the precipitate once by decantation. An addition of 5 c.c. of the basic lead acetate solution is next made, and the shaking repeated. Then, after two to three minutes standing, the liquid is shaken with 5 c.c. of the sodium sulphate solution, made up to 250 c.c., shaken, and filtered. The first 8 to 10 c.c. are rejected, and 25 c.c. of the remaining filtrate are treated with 3 to 4 drops of dilute sulphuric acid (1 : 5) to precipitate the last traces of lead, and then with 4 grms. of pure potassium dichromate and about 25 c.c. of water. The flask is shaken until the salt has dissolved, after which 50 c.c. of dilute sulphuric acid (1 : 1) are added and the liquid heated for fifteen minutes as described above. After cooling, 4 grms. of pure ferrous ammonium sulphate are added, and the excess of this salt subsequently titrated with dilute potassium dichromate solution (7·456 grms. per litre). A sample showing 85·10 per cent. of glycerol in the first test gave 84·74 per cent. in the second estimation.

Amount of Crude Glycerine to be taken for Second Estimation.

Glycerol found in Preliminary Test. Per Cent.	Amount of Sample to be used. Grms.	Amount reacting with Dichromate. Grm.	Glycerol found in Preliminary Test. Per Cent.	Amount of Sample to be used. Grms.	Amount reacting with Dichromate. Grm.
100	20·00	0·5000	87	22·98	0·5745
99	20·22	0·5055	86	23·24	0·5810
98	20·44	0·5110	85	23·50	0·5875
97	20·66	0·5165	84	23·80	0·5950
96	20·88	0·5220	83	24·10	0·6025
95	21·10	0·5275	82	24·40	0·6100
94	21·32	0·5330	81	24·70	0·6175
93	21·54	0·5385	80	25·00	0·6250
92	21·76	0·5440	79	25·32	0·6330
91	21·98	0·5495	78	25·64	0·6410
90	22·20	0·5550	77	25·96	0·6490
89	22·46	0·5615	76	26·28	0·6570
88	22·72	0·5680	75	26·60	0·6650

Glycerol in Soap Lyes.—About 25 grms. are diluted to 50 c.c., neutralised with acetic acid, treated with basic lead acetate solution till no further turbidity is produced, and then with the same volume of the sodium sulphate solution, and made up to 100 c.c., shaken, and filtered. The glycerol is estimated in 10 c.c. of the filtrate as described above, and a second estimation is made upon a quantity of the sample corresponding to the result obtained (see table). This quantity (corresponding to about 0·5 grm. of pure glycerol) is diluted, neutralised with acetic acid, shaken with silver carbonate, then with basic lead acetate solution (added in portions of 0·5 c.c. until no further precipitate results), and with a quantity of sodium sulphate solution equal to the lead solution used, after which it is made up to 100 c.c. and filtered. The glycerol is then estimated as described for crude glycerol.

Amount of Soap Lye to be taken for Second Estimation.

Glycerol found in Preliminary Test.	Amount of Sample to be used.	Amount reacting with Dichromate.	Glycerol found in Preliminary Test.	Amount of Sample to be used.	Amount Reacting with Dichromate.
Per Cent.	Grms.	Grms.	Per Cent.	Grms.	Grms.
5·0	40·00	10·00	13·0	15·38	3·84
5·5	36.36	9·09	13·5	14·81	3·70
6·0	33·33	8·33	14·0	14·28	3·57
6·5	30·77	7·69	14·5	13·80	3·45
7·0	28·57	7·14	15·0	13·33	3·33
7·5	26·66	6·66	15·5	12·90	3·22
8·0	25·00	6·25	16·0	12·50	3·12
8·5	23·53	5·88	16·5	12·12	3·03
9·0	22·22	5·55	17·0	11·76	2·94
9·5	21·05	5·26	17·5	11·42	2·85
10·0	20·00	5·00	18·0	11·11	2·78
10·5	19·05	4·76	18·5	10·82	2·70
11·0	18·18	4·54	19·0	10·52	2·63
11·5	17·39	4·35	19·5	10·26	2·56
12·0	16·66	4·16	20·0	10·00	2·50
12·5	16·00	4·00			

C. A. M.

Note by Otto Hehner.—Neither the dichromate nor the acetin method gives a correct result in the case of some impure glycerines, unless the dichromate or acetyl value of the non-volatile impurity is determined and deducted.

Report of the [Dyestuffs] "Fastness Committee." (*Chem. Zeit.*, 1914, **38**, 154–158.)—A committee representing the principal German dyestuff manufacturers, consumers, and other interests, has adopted a set of standard methods and typical dyestuffs for testing and classifying dyeings on cotton and wool according to their resistance to various influences. The fastness to light is divided into eight classes; the other properties are each classified in five degrees of resistance. As types for the various groups, only those dyestuffs which are generally available have been selected, and separate sets of standards are provided for cotton and wool. The properties standardised include fastness to light, washing, water, rubbing, ironing, sulphuring, sweat, alkali, boiling with acid potassium tartrate, cold mineral and organic acids, prolonged boiling with alkali ("bowking"), hypochlorites (cotton), mercerisation (cotton), peroxides (wool), "fulling" (wool), "carbonising" (wool), "potting" (wool), steaming (wool), sea-water (wool). The manner of applying the tests and the manner of dyeing the type standards are described in detail in each case.

J. F. B.

Analysis of Waste Acids from Nitrocellulose Manufacture. T. Chandelon. (*Bull. Soc. Chem. Belg.*, 1914, **28**, 58.)—Oxalic acid may be present, especially in repeatedly used nitrating baths, and, by giving a fictitious value to the nitrous acid, determined by titration with permanganate, give rise to a low nitric acid value which is determined by difference from the combined nitric and nitrous acid. The oxalic acid may be determined by neutralising the acid with ammonia, boiling, to destroy nitrous acid, cooling, and titrating with permanganate. Other substances which reduce permanganate at temperatures above 40° C. are present in the waste acids. O. E. M.

Heat Test of Explosives. A. C. Egerton. (*J. Soc. Chem. Ind.*, 1914, **33**, 113.) —The view that equilibrium is reached in the heat-test tube is supported by the constancy of the heat-test time with varying numbers of papers or drops, and in continuous tests. Other explanations of this behaviour are considered and rejected; reduction of nitric peroxide to nitrogen during a continuous test does not account for it, for experiments in a special apparatus based on Ramsay's device for measuring small quantities of gases show that only about one-sixtieth of the theoretical quantity of nitrogen, given off as oxides of nitrogen or as nitrogen, is evolved from guncotton heated to 80° C., and does not increase on prolonged heating. At this temperature the explosive behaves in part like an ordinary dissociable substance, but simultaneously undergoes an irreversible decomposition. Other points arising out of the former paper are dealt with, and the advantages of simultaneous continuous tests of the sample and of a standard explosive are pointed out (*cf.* ANALYST, 1913, **38**, 275).

O. E. M.

Estimation of Hydroxybutyric Acid. P. A. Shaffer and W. M. Marriott. (*J. Biol. Chem.*, 1913, **16**, 265; through *Chem. Zentralbl.*, 1914, I., 821.)—Estimation of β-hydroxybutyric acid by oxidation with chromic acid to acetone gives about 90 per cent. of the true value. The acid is isolated from the ether extracts as the double calcium zinc salt, which crystallises from a mixture of solutions containing equivalent quantities of the two salts in needles or needle-like plates melting about 240° C. with decomposition; solubility in water, 1 : 10; insoluble in alcohol; $[\alpha]_{D20}$ – 16·26°. The oxidation method gave results agreeing with those obtained by Black (*J. Biol. Chem.*, 1908, **5**, 209) by polarisation of the ethereal extract, but the presence of other lævorotatory substances must be guarded against. O. E. M.

Detection of Methyl Alcohol and Ethyl Alcohol. J. J. Blanksma. (*Chem. Weekblad*, 1914, **11**, 26-29; through *Chem. Zentralbl.*, 1914, I., 574.)—The method proposed depends on a reaction between sodium methoxide or sodium ethoxide and 1-chloro-2 : 4-dinitrobenzene, according to the equation—

$$C_6H_3Cl(NO_2)_2 + NaOC_2H_5 = C_6H_3{\cdot}OC_2H_5(NO_2)_2 + NaCl.$$

This reaction has been described previously by Lulofs (*Rev. trav. Chim. Pays-Bas*, 1901, **20**, 292). In carrying out the test, the liquid under examination is neutralised with magnesium oxide or calcium carbonate, then treated with a small quantity of *p*-nitrophenylhydrazine and submitted to distillation, a fractionating column being employed. Twenty-five c.c. of distillate are collected, and a determination of the sp. gr. gives the quantity of alcohols present, and from this quantity the amount of chlorodinitrobenzene to be added is calculated. The alcoholic liquid is now concentrated by repeated distillation until it contains from 90 to 95 per cent. of alcohol. To this concentrated alcoholic solution are added 12 mgrms. of sodium and 100 mgrms. of chlorodinitrobenzene, the mixture is warmed for a short time, then diluted with water, and the 1 : 2 : 4-dinitrophenetole (m.-p., 85·2° C.), or 1 : 2 : 4-dinitroanisole (m.-p., 86·9° C.), formed, respectively, is dried. The alcohol present is identified by mixing the substance thus obtained with pure dinitrophenetole or dinitroanisole, and determining the m.-pt. of the mixture; for instance:

Molecular percentages 1 : 2 : 4-dinitrophenetole ...	0	9·2	27·6	38·4	46·2	56·4	65·6	80·5	100 %
M.-pt. of the mixture with dinitroanisole	86·9	81·8	76·0	61·5	53·0	54·0	66·2	75·0	85·0 °C.

W. P. S.

Determination of the Hydrocarbon Content of Concentrated Lemon Oils. E. Böcker. (*J. prakt. Chem.*, 1914, **89**, 199-207; through *J. Soc. Chem. Ind.*, 1914, **33**, 275.)—Ten c.c. of the oil are measured into a cassia flask and the citral determined by shaking with freshly-prepared sodium sulphite solution according to Burgess' method (*J. Soc. Chem. Ind.*, 1901, 1178). If the amount of undissolved oil is small, the citral is removed in the same way from another 5 or 10 c.c. of the oil. Five c.c. of the oil freed from citral are transferred to a stoppered funnel of 600 to 700 c.c. capacity containing 500 c.c. of 51 per cent. alcohol (by volume) cooled to − 2° to 0° C. The whole is thoroughly shaken when the aromatic portions of the oil pass into solution leaving the hydrocarbons practically undissolved. The funnel is inverted, kept for eight to ten hours in ice-water at 0° C., carefully returned to the upright position and allowed to stand till the alcoholic portion has become clear, which may take from twelve to forty-eight hours. The alcoholic portion is drawn off and the oil (hydrocarbons) transferred to a small measuring cylinder, any drops adhering to the funnel being washed down with a little 51 per cent. alcohol. Larger quantities of the original oil can be fractionated at reduced pressure to separate terpenes and sesquiterpenes. The citral in the separate fractions is determined as above and the undissolved oil from each treated with a corresponding volume of 51 per cent. alcohol so as to ascertain the relative proportions of terpenes and sesquiterpenes.

Viscosity of Oils. J. L. Strevens. (*J. Soc. Chem. Ind.*, 1914, **33**, 109.)—The absolute viscosity values for a number of oils (Dunstan and Strevens, *J. Soc. Chem. Ind.*, 1912, **31**, 1063) are corrected for a new viscometer constant calculated from the viscosity values of phenol determined by Dunstan and Thole (*ibid.*), and extended to other vegetable and animal oils, solid fats, and waxes. The old values below 88° C. are high, and those above are low. The previous viscosity-temperature curves are materially altered, especially in the high temperature region. In general, viscosity is increased by a rise in molecular weight, formation of hydroxyl groups, polymerisation, solution of solids in the oil, and by high molecular weight and conjugated double bonds, and diminished by unsaturation. The determination of viscosities at certain standard temperatures should be superseded by the construction of a viscosity-temperature curve over a range of 15° to 100° C. for ordinary lubricants, and to much higher temperatures for cylinder and gas-engine oils.

O. E. M.

Pyridine as a Solvent in the Estimation of Hydroxyl Groups by Means of Alkyl Magnesium Halides. A. P. Tanberg. (*J. Amer. Chem. Soc.*, 1914, **36**, 335-337.)—Zerewitinoff (ANALYST, 1911, **36**, 117; 1912, **37**, 571) has recommended the use of commercial pyridine as a solvent when employing the Grignard reaction for the estimation of hydroxyl groups in organic compounds, and claims to have obtained accurate quantitative results when using it. The author fails entirely to confirm this, either when working with ordinary or purified pyridine. Thus, with *a*-naphthol over twice the theoretical yield of methane was obtained, and it was found, after taking all precautions to insure the absence of moisture, that considerable amounts of gas (presumably methane) were immediately liberated when various samples of high-grade dry pyridine were mixed with methyl magnesium iodide in the absence of any substance containing an hydroxyl group. The fact that Oddo (ANALYST, 1911, **36**, 459) obtained satisfactory results may have been due to the fact that he employed magnesium ethyl iodide. H. F. E. H.

Colour Reaction of Succinic and Malic Acids. W. O. de Coninck. (*Bull. Soc. Chim.*, 1914, **15**, 93-94.)—On heating calcium salicylate with a concentrated aqueous solution of succinic acid, there is produced a stable rose coloration which may be exposed to direct sunlight for several days without fading. Malic acid gives a similar coloration, but if the liquid is boiled for fifteen to twenty minutes, the colour first darkens and eventually disappears, leaving a yellow or light brown liquid. It is possible in this way to distinguish between the two acids.

C. A. M.

INORGANIC ANALYSIS.

Analysis of Golden Sulphide of Antimony. W. Schmitz. (*Gummi-Zeitung*, 1913, **28**, 453-455: through *Chem. Zentralbl.*, 1914, I., 495-496.)—A method described by Utz (*Gummi-Zeitung*, 1913, **28**, 126) is shown to give results which are always at least 1·5 per cent. too high, and which may be 3 per cent. too high. The following method is recommended. It depends on reduction to the antimonious condition and titration with bromate. Solution and reduction are effected by digestion with hydrochloric acid of sp. gr. 1·19, the solution being finally heated until hydrogen sulphide is no longer evolved. Tartaric acid is added, and the solution is diluted and filtered through a filter that will retain finely-divided sulphur. A little more hydrochloric acid is added, and the solution titrated with potassium bromate solution, either in the cold with methyl orange as indicator, or with indigo at a higher temperature. The bleaching of the indicator affords a very sharp end-point.

$$2KBrO_3 = 3Sb_2O_3 = 3Sb_2S_5.$$

Potassium bromate solution does not alter in strength in six months. G. C. J.

Detection of Bromides in Presence of Thiocyanates and Ferrocyanides. Icilio Guareschi. (*Atti. R. Accd. Sci. Torino*, 1914, **49**, 15; through *Chem. Zentralbl.*, 1914, I., 815.)—Thiocyanates and ferrocyanides are destroyed by the addition of chromic acid in the solid form or in strong solution. The liberated bromine is carried by a slow stream of air over paper saturated with fuchsin-sulphurous acid, or with Hoffmann's violet decolorised with sulphur dioxide. The reaction is sensitive to 0·0003 grm. of bromine in 0·5 grm. of potassium thiocyanate, to 0·001 grm. of potassium bromide in 1 grm. of potassium ferrocyanide or ferricyanide, or to 1 per cent. of silver bromide in potassium ferrocyanide. Bromine may be readily detected in Prussian blue and ultramarine. Potassium permanganate cannot be substituted for chromic acid; ammonia is set free, and may mask the reaction. Cyanogen bromide is oxidised by chromic acid, even in dilute solution, with formation of carbon dioxide, ammonia, and bromine. O. E. M.

Estimation of Blast-Furnace Slag in Portland Cement. F. Hart. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1914, **27**, 286-290.)—The method proposed depends on the difference between the specific gravities of blast-furnace slag and Portland cement, and also on the different proportions of sulphides present in the two materials. The slag has a sp. gr. of 2·830 to 2·885, and contains from 1·1 to 1·27 per cent. of sulphide sulphur; whilst the cement has a sp. gr. of 2·887 to 3·185 (it generally exceeds 3·05), and contains from 0 to 0·14 per cent. of sulphide sulphur. It may be mentioned that when Portland cement is heated to a high temperature the sp. gr. rises to 3·106 to 3·247, but ignition diminishes the sp. gr. of slag to 2·128 to 2·778. The estimation of furnace slag in cement is carried out as follows: The sample is passed through a sieve of 5,000 meshes to the sq. cm., and freed from particles of iron by means of a magnet. Twenty grms. of the fine powder are then mixed in a separating-funnel with a mixture of benzene and methylene iodide having a sp. gr. of 3·00, and the air is exhausted from the separator in order to remove air-bubbles from the mineral particles; the mixture is then agitated from time to time for two hours, and allowed to remain at rest for one hour. The heavier portion is now collected on a filter, and the lighter portion on another filter; both portions are washed with benzene and dried in a steam-oven. The heavier portion is then shaken with a benzene-methylene iodide mixture of sp. gr. 3·05, and the lighter portion with a mixture of sp. gr. 2·96; after separation the lighter portions are treated with a mixture of sp. gr. 2·70. In this way the following fractions are obtained: (1) Heavier than 3·05, cement; (2) between 3·0 and 3·05, mixture of cement and slag; (3) between 2·96 and 3·0, mixture of cement and slag; (4) between 2·78 and 2·96, blast-furnace slag; (5) lighter than 2·70, coke, ash, calcium sulphate, etc. The fractions (1) and (4) are examined microscopically, and if they do not appear to consist of cement and slag respectively they are submitted to further treatment with the benzene-methylene iodide mixtures. The percentage quantities of sulphide sulphur are then estimated in these two fractions, (1) and (4), as well as in the original material, and the amount of blast-furnace slag present is calculated from the results obtained. For instance, suppose the original material contained 0·26 per cent., fraction (1), 0·02 per cent., and fraction (4), 1·10 per cent. of sulphide sulphur, then the quantity of slag present in the sample will be $100\frac{0·26-0·02}{1·10-0·02}$, or 22·22 per cent. W. P. S.

Reduction Method of Analysing Nitrates, Chlorates, and Peroxides.

C. Russo and G. Sensi. (*Gazz. Chim. Ital.*, 1914, **44**, 9-15.)—The method of titrating ferric salts in the presence of methylene blue as indicator may be adapted to the analysis of other oxidising compounds. *Nitrates.*—The reduction is made in a large, closed flask, connected by means of a tube bent twice at right angles with a second smaller flask, also closed, in which is placed 120 c.c. of water. A current of carbon dioxide or other inert gas is passed through the flasks, and, after expulsion of all air, a solution of decolorised methylene blue (made by adding 2 drops of methylene blue solution to 2 to 3 c.c. of water, and boiling the liquid with 1 drop of $\frac{N}{5}$ stannous chloride solution) is introduced into the smaller flask, whilst about 3 grms. of ferrous ammonium sulphate, the weighed quantity of, *e.g.*, potassium nitrate, and 10 c.c. of concentrated hydrochloric acid are placed in the large flask. The flask is gently heated until the oxidation is complete and the nitrogen peroxide has been expelled by the carbon dioxide. The water in the second flask is also brought to boiling-point before completion of the oxidation, and, after stoppage of the current of gas, it is drawn back into the large flask, the contents of which are immediately titrated with $\frac{N}{5}$ stannous chloride solution, previously standardised upon $\frac{N}{10}$ ferric chloride solution prepared from pure ferric oxide. The percentage of nitrate is calculated by means of the formula $x=\frac{MVC}{30\ P}$, where C represents the concentration (grm. equivalents) of the stannous chloride solution, V the volume in c.c. of the stannous chloride solution required by the weight, P, of the nitrate, and M the molecular weight of the nitrate. *Chlorates.*—The estimation is made in a similar manner by oxidising ferrous ammonium sulphate and titrating the ferric salt produced, but in this case the presence of air does not interfere with the accuracy of the results. The solution of the chlorate is boiled with the excess of the ferrous salt, and 10 c.c. of hydrochloric acid added in small portions at a time until the oxidation is complete. *Peroxides.*—A weighed quantity of, *e.g.*, manganese dioxide, or lead peroxide, is boiled with about 100 c.c. of water (previously freed from air), an excess of ferrous ammonium sulphate added, and then 10 c.c. of hydrochloric acid, the boiling continued until a clear solution is obtained, and the liquid titrated with stannous chloride solution. C. A. M.

Application of Sodium Paratungstate and the Blowpipe Flame to the Estimation of the Acid Radicals in Chlorides, Chlorates, Perchlorates, Bromides, Bromates, and Fluorides.

S. B. Kuzirian. (*Zeitsch. anorg. Chem.*, 1914, **85**, 118-126.)—Though carbonates, nitrates, iodides, and iodates are readily decomposed by fusion with sodium paratungstate, the salts named in the title of this paper are so slowly acted on that the method has not hitherto been available for their estimation. That chlorides and bromides should be very slowly attacked might be expected, as the reaction must depend on the action of atmospheric oxygen or moisture. Chlorates and bromates might be expected to react more readily, and they do so, but they are largely converted into chloride or bromide in the earlier stages of heating, and these salts are only decomposed very slowly. It is now found that superheated steam readily supplies the necessary oxygen, and that a small blowpipe flame directed on the surface of the assay is a suitable supply of superheated steam. A platinum crucible is charged with the reagent, tared, the substance to be analysed added, the crucible and its contents weighed again, then heated over a small flame until any violent reaction, as with chlorate, is over, and finally heated from above with a blowpipe flame for about ten minutes. The reagent is prepared by fusing together equal weights of sodium tungstate and tungstic acid. G. C. J.

New Method of Titrating Ferric Salts. C. Russo. (*Gazz. Chim. Ital.*, 1914, **44**, 1-8.)—The method is based upon the sharp reduction of methylene blue by means of stannous chloride in the presence of dilute (10 per cent.) hydrochloric acid. The solution of the ferric salt is acidified with hydrochloric acid, or, if too acid, is partially neutralised with ammonia, and is then treated with a few drops of a 1 to 1·2 per cent. solution of methylene blue, and boiled until all air is expelled. It is next treated with standard (approximately $\frac{N}{5}$) stannous chloride solution up to a point just before the green colour of the solution changes to blue, then boiled, and the addition of the stannous chloride continued, drop by drop, until the blue coloration becomes pale, after which it is again boiled, and the titration completed. The titration may be repeated after oxidising the reduced ferrous salt with hydrogen peroxide. By combining the method with the ordinary permanganate method, the amounts of ferric and ferrous iron in a mixture of salts may be rapidly and accurately estimated. C. A. M.

Analysis of Monazites. S. J. Johnstone. (*J. Soc. Chem. Ind.*, 1914, **33**, 55-59.)—The title of this paper is "Monazites from Some New Localities," but, in support of his analytical numbers, the author describes his methods in detail, and includes some test numbers obtained with known quantities of thoria, ceria, lanthana, yttria, and phosphoric acid. These test-numbers are very satisfactory. None of the methods employed is less than ten years old, but the way in which they are combined has points of originality, and finds justification in the excellent test numbers obtained. G. C. J.

Estimation and Distinction of Ozone, Nitrogen Peroxide and Hydrogen Peroxide at High Dilutions. J. N. Pring. (*Chem. News*, 1914, **109**, 73-75.)—The object of the research was to establish methods for the estimation of ozone, nitrogen peroxide, and hydrogen peroxide in such concentrations as may occur in the upper strata of the atmosphere. At high dilutions (6 parts per million) the sole product of the reaction of ozone or of hydrogen peroxide with neutral potassium iodide solution appears to be potassium hypoiodite. The two gases can be distinguished by the action of hydrogen peroxide on titanium sulphate. At higher concentrations than that named, the reaction between ozone and potassium iodide is more complex, more or less iodate (and periodate) being formed; but the proportion of iodate formed is negligibly small even in the comparatively high concentration of 0·01 per cent., a concentration fifty times greater than that which imparts a distinctly perceptible odour to air. Hydrogen peroxide never gives rise to iodate, but if in high concentration it is progressively destroyed by the iodide and hypoiodite with liberation of oxygen, the amount of hypoiodite rapidly reaches a maximum beyond which it does not increase. Nitrogen peroxide, even in the highest dilutions, gives rise to iodate as well as hypoiodite when brought into contact with potassium iodide. This behaviour, as well as the fact that after acidification and titration with thiosulphate, iodine persistently reappears, serves to distinguish nitrogen peroxide from the other gases.

G. C. J.

Nitrogen and Chlorine in Rain and Snow. G. H. Wiesner. (*Chem. News*, 1914, **109**, 85-87.)—Thirty-one samples in all were tested, twenty-two of rain and nine of snow; these were collected between February 22 and June 5, 1912, at Mount Vernon, U.S.A., a village of 1,700 inhabitants, without manufacturing industries. The rainfall during this period was 15 inches and the snow 24 inches, equivalent to 2 inches of rain—an amount above the normal for the district.

PARTS PER MILLION (AVERAGE).

	Snow.	Rain.
Free ammonia	3·35	0·931
Albuminoid ammonia ...	3·84	1·13
Nitrite nitrogen	0·0021	0·0018
Nitrate „	0·19	0·15
Chlorine	4·7	4·8

H. F. E. H.

Dehydration and Separation of Silica. F. A. Gooch, F. C. Reckert, and S. B. Kuzirian. (*Zeitsch. anorg. Chem.*, 1914, **85**, 23-30.)—It is found that the difficulty of igniting silica to constant weight is not due to the obstinate retention of water but to the fact that, at the temperature of the blowpipe flame, the solid impurities are slowly volatilised or partially decomposed. In silica separated from a silicate in the usual manner, the chief impurity is sodium chloride, which is partly volatilised but partly decomposed with loss of chlorine and formation of sodium silicate. When the silica is finally treated with hydrofluoric and sulphuric acids, any sodium sulphate remaining should be calculated to sodium oxide, and this amount substracted from the weight of crude silica. The amount of silica passing into the filtrate in an ordinary silica separation depends on the exact manner in which the dehydration is effected. When effecting this at 110° C., the authors find the average loss of silica about 1 per cent. The loss cannot be reduced by repeated evaporation, but evaporation of the filtrate leads to the recovery of all but a negligible trace.

G. C. J.

Correcting Silica for Occluded Salts. S. B. Kuzirian. (*Zeitsch. anorg. Chem.*, 1914, **85**, 430-434.)—The impurities actually present in a silica precipitate before ignition may include ferric oxide, alumina, and titanium oxide, resulting from the decomposition of the corresponding chlorides during the operation of rendering the silica anhydrous and insoluble; sodium chloride will nearly always be present in appreciable amount, and other chlorides may be. After prolonged ignition over a blowpipe flame, chlorides will be present only in negligible traces, but the bases originally combined with chlorine will now be combined with silica, with the exception that sodium chloride is partially volatilised as such, and only partially decomposed by the silica. When the ignited precipitate is treated with hydrofluoric and sulphuric acids and the residue ignited in the usual way, iron oxide is recovered as such, but soda, which may be the chief impurity, is recovered as sulphate. A better method of dealing with a silica precipitate is to ignite it over an ordinary Bunsen flame, to decompose chlorides by addition of a few drops of sulphuric acid, the excess of which is cautiously expelled, to weigh the silica and treat it with hydrofluoric and sulphuric acids as usual. The weight of the residue thus obtained should be the exact measure of the impurities weighed with the silica.
G. C. J.

Physico-Chemical Estimation of Sulphates. A. Kling and A. Lassieur. (*Comptes rend.*, 1914, **158**, 487-489.)—The statement made by Bruno and d'Auzay (ANALYST, 1912, **37**, 282) that this method yields inaccurate results when applied to the estimation of sulphates in wine, is confirmed by the authors. The low results obtained appear to be due to the occlusion of alkali sulphates by the barium sulphate; the loss becomes more marked as the quantity of alcohol present increases, and is greater in the case of potassium sulphate than in that of other alkali sulphates.
W. P. S.

Solubility of Silica and Rendering Silica Insoluble in Acid Solutions. M. Hunder and A. Suleimann. (*Ann. Chim. anal.*, 1914, **19**, 45-49.)—It is found that when solutions of sodium silicate are acidified with hydrochloric acid, evaporated to dryness, the residue baked for two hours at 110° C., and then treated with a little hydrochloric acid and the silica washed, ignited, and weighed in the usual manner, there is an average loss of 0·7 per cent. More than 90 per cent. of this lost silica can be recovered by a single evaporation of the filtrate and washings. Repeated evaporations before the first filtration do not diminish the loss with the first filtrate. When nitric acid is used instead of hydrochloric acid, the loss of silica with the first filtrate is rather less; the whole of it can be recovered by a single evaporation of the filtrate. There is a tendency to high results when using nitric acid, as any sodium nitrate not wholly removed by washing is only partly decomposed on igniting the silica, whereas traces of sodium chloride are wholly volatilised. With sulphuric acid as much as 4 per cent. of the total silica may pass into solution, but this can be recovered by evaporating the filtrate. When this is done, the total result may be slightly high, due to the difficulty of washing out the last traces of sodium sulphate. The experiments were all made with 0·5 grm. pure silica and 6 grms. sodium carbonate, which were fused together and then treated in the usual manner in which the products of silicate fusions are treated.

The solubility of precipitated silica in acid solutions was determined with the following results, which represent grms. dissolved by 1,000 c.c. of the solvent:

Solvent.	Sp. gr.	Solubility at 100° C.	Solubility at 18° C.
Hydrochloric acid	1·06	0·12	0·03
,, ,,	1·09	0·14	0·03
,, ,,	1·12	0·33	0·05
Nitric acid	1·13	0·10	0·02
,, ,,	1·20	0·08	0·03
,, ,,	1·27	0·04	0·03
Sulphuric acid	1·40	0·02	0·04

Cf. Archbutt (*J. Soc. Chem. Ind.*, 1892, **11**, 215) and Hillebrand (ANALYST, 1902, **27**, 289).

G. C. J.

Tellurium Dioxide for Combustion Analyses. R. Glauser. (*Chem. Zeit.*, 1914, **38**, 187.)—Tellurium dioxide at a red heat is a mobile, heavy liquid which has a powerful oxidising action upon many bodies not otherwise easily attacked. Small globules of metallic tellurium are produced and float about in the melt. If the product of oxidation is a gas—*e.g.*, carbon dioxide or nitrogen—it may be collected and estimated, and the tellurium and tellurium dioxide are so little volatile that there is no difficulty in retaining them before the gas is absorbed. The fused tellurium dioxide employed in excess is a good solvent for non-volatile basic products, and is very suitable for the combustion of elementary carbon, carbides, cyanamides, and nitrides, which are combustible with difficulty. The author has tested the method for the estimation of nitrogen in aluminium nitride, of carbon and nitrogen in calcium cyanamide, of carbon in ferro-chromium, and certain hard steels which can only be ground with difficulty but which easily dissolve in fused tellurium dioxide, even in comparatively large lumps. The time occupied in making a combustion with fused tellurium dioxide is generally quite short, and the results as a rule are in good concordance with those obtained by ordinary methods. The method may possibly be developed for the estimation of sulphur in ores, etc. The author prepares the tellurium dioxide from the metal, and the greater part may be recovered after use.

J. F. B.

Effect of Tungsten on Ammonium Molybdate Assay for Lead. H. Lavers. (*Chem. News*, 1914, **109**, 97.)—Some Broken Hill lead concentrates contain both wolfram and scheelite, the amount of which may correspond to 5 or 6 per cent. of tungstic oxide. As the presence of these minerals in Broken Hill ore has only recently been reported, the custom has been for all concentrates to be bought and sold on the basis of a volumetric estimation of lead by the molybdate method. With this method of estimation, as usually carried out, the presence of tungsten interferes, tungstic acid being converted into ammonium tungstate by the ammonium acetate used to dissolve the lead sulphate, and this ammonium tungstate then reacts with the lead exactly as molybdate does. With 5·5 per cent. tungstic acid in the concentrate, the lead will be underestimated by 3 per cent.

Lead ores containing tungsten minerals should be treated with hydrochloric and nitric acids, the solution evaporated to low bulk, diluted, and filtered from silica and tungstic acid. The filtrate is evaporated with sulphuric acid until dense white fumes appear, and the molybdate assay then completed in the usual manner. This method is now embodied in many contracts for the sale of Broken Hill ore. G. C. J.

Estimation of Water of Crystallisation in Sulphates. S. B. Kuzirian. (*Zeitsch. anorg. Chem.*, 1914, **85**, 127-132.)—The water of crystallisation of aluminium sulphate, the alums, and many other sulphates which lose sulphuric anhydride on heating alone, can be estimated exactly by heating with neutral sodium tungstate, the soda of which combines with any sulphuric anhydride which may be liberated. The estimation is most conveniently conducted by weighing the sulphate into a crucible containing some dry sodium tungstate, heating to fusion, maintaining in this state for fifteen to twenty minutes, allowing to cool in a desiccator and reweighing. Identical results are obtained by conducting the fusion in a platinum boat in a combustion-tube, using a current of dry air to sweep the moisture into an absorption-tube. The reagent is prepared from the commercial salt, which always contains carbonate, by fusing it and adding tungstic acid in small portions until there is no further effervescence. The presence of excess of tungstic acid—that is, of paratungstate—is of no consequence, but the product must be free from any trace of carbonate.

G. C. J.

APPARATUS, ETC.

Fire-Damp Whistle. F. Haber. (*J. Soc. Chem. Ind.*, 1914, **33**, 54.)—The fire-damp whistle is an instrument for detecting the presence of fire-damp by acoustic means. It consists of a brass cylinder 25 cms. long and 6 cms. in diameter. Inside the brass cylinder are two small pipes, which are tuned to give the same note in air. They are blown by one and the same current of air which is provided by a hand pump, which is simply the outer sleeve of the brass cyclinder. Both pipes are provided with membranes, which prevent the operating air current from entering the pipe-tubes (resonators). The space beyond the membrane forms, in the case of each pipe, therefore, a closed gas chamber, the nature of the gas contained in which determines the pitch of the note given by the pipe. In the case of one pipe, the gas chamber is filled with pure air outside the mine, this air remaining unchanged during the sojourn in the mine. Into the closed chamber of the other pipe can be brought the air of the mine, purified from dust, moisture, and carbon dioxide by a cartridge of soda lime. If the mine air contains methane, then, on sounding the two pipes by means of the sleeve-pump, beats are heard, whose frequency increases with the proportion of methane in the air of the mine. The beating of the notes produces a characteristic "trill" when the methane content approaches the explosive limit.

G. C. J.

Quantitative Sublimation. E. Philippe. (*Mitt. Lebensmitteluntersuch. u. Hyg.*, 1913, **4**, 351-365; through *Chem. Zentralbl.*, 1914, I., 576.)—The following procedure is recommended for the estimation of caffeine in coffee by means of the sublimation apparatus described previously by the author (ANALYST, 1912, **37**, 428): Five grms. of the finely ground coffee are moistened with 10 c.c. of ammonia and extracted repeatedly with chloroform. The chloroform extract is filtered, evaporated, and the residue is dissolved by warming with dilute sulphuric acid. After cooling, the solution is extracted with ether to remove fat, then rendered ammoniacal, and the caffeine is shaken out with chloroform. The residue obtained on evaporating the chloroform solution is then sublimed in the apparatus. W. P. S.

Estimation of the Weight of a Substance by Titration. H. Dubovitz. (*Chem. Zeit.*, 1914, **38**, 222.)—The weight of a substance may sometimes be calculated from the results of fractional titration of a constituent therein. For example, in estimating the weight, a, of an unknown quantity of oil, the acid-value, s, is first determined, showing that s kilos of potassium hydroxide are required to neutralise 1,000 kilos of oil. A quantity of potassium hydroxide (v kilos) insufficient to neutralise the whole of the oil is added, and the quantity of unneutralised oil will then correspond to $a - 1{,}000\ v/s$, whilst $1{,}000\ v/s$ kilos will have been neutralised. The acid-value, s_1, is again estimated, and the oil still unneutralised will require $x = a\ s_1/1{,}000$ kilos. of potassium hydroxide for neutralisation. But the unneutralised oil was shown before to correspond to $a - 1{,}000\ v/s$, for the neutralisation of which the kilos of potassium hydroxide required would be—

$$w = \frac{a\ s - 1{,}000\ v}{1{,}000}.$$

And since $x = w$,

$$\frac{a\ s - 1{,}000\ v}{1{,}000} = \frac{a\ s_1}{1{,}000};$$

whence the required weight of the oil is—

$$a = \frac{1{,}000\ v}{s - s_1}.$$

Note.—It is assumed that the amount of alkali neutralised does not vary, whether the fatty acid is wholly or partly neutralised.

C. A. M.

Uses of the Concentrated Filament Tungsten Lamp in the Laboratory. F. A. McDermott. (*J. Amer. Chem. Soc.*, 1914, **36**, 454-455.)—The specification of the lamp described is as follows: Mazda, stereopticon type (concentrated filament), 100 watt, 110 volt, G-30 bulb, unskirted. The lamp gives about 60 candle power and the filament is formed into a close coil which is looped over supports about 1 cm. apart, the loops forming an open cylinder about 1 cm. in diameter. The bulb is a round one about 7·5 cms. in diameter. The lamp is of use for reading highly coloured solutions in the polariscope, the penetration intensity being high, and it has the advantage of giving no bright-line images of portions of the filament in the field of the instrument. H. F. E. H.

GOVERNMENT REPORT.

Report on the Work of Inspectors of Foods for the Year 1912-1913. A. W. J. McFadden. (Extracted from the Annual Report of the Medical Officer of the Local Government Board.) As in former years (*cf.* ANALYST, 1913, **38**, 131) the Report is divided as follows:

1. *Work in Special Relation to the Public Health (Regulations as to Food) Act*, 1907.—Improvement has been maintained in the character of meat imported from abroad, especially in the case of boxed meat ; this now consists of various well-defined cuts which are frozen separately before being placed in the packing-case, so that each piece is removable for examination on arrival in this country. The imports of Chinese pork and bacon showed a decrease; one carcass was condemned on account of pyæmia, five on account of abscesses, eight for tuberculosis, and three for hæmorrhagic adenitis. Of 223 cases of Chinese bacon and hams, two contained excessive quantities of boric acid. A large number of Siberian tongues were found to show lesions of foot-and-mouth disease, and during the early part of the year many cases of tuberculous lesions were detected in consignments of chilled Argentine beef. Except in one case, imported chilled meat which had been treated with formaldehyde did not contain an excessive amount of this preservative. Forty-two samples of milk arriving from the Continent were examined; all the milk appeared to have been pasteurised and tubercle bacillus was not found in any of the samples.

It is anticipated that attempts to evade the requirements of the Milk and Cream Regulations (*cf.* ANALYST, 1912, **37**, 109), which came into force on October 1, 1912, will be of comparatively rare occurrence if active steps are taken by the local authorities.

The Board's attention has been drawn to circulars sent to butter and margarine merchants and to milk dealers offering for sale a new preservative called "Suffectus." "Suffectus M." was found to consist of an aqueous solution of potassium fluoride (18·2 per cent.), with a small proportion of potassium carbonate; "Suffectus F. C." (for cream) was a white powder, consisting of potassium fluoride (37·2 per cent.) and lactose (50·5 per cent.), whilst "Suffectus F. B." (for butter) contained potassium fluoride (39·8 per cent.) and sodium chloride (51·5 per cent.). Two other fluoride preservatives, "Purit" (chiefly sodium fluoride) and "Extra Purit" (sodium fluoride and sodium benzoate), were brought under the notice of the Board. The suppression of the use of such substances in milk and cream should offer no difficulty, and proceedings have been instituted in several cases.

2. *Local Arrangements for Food Inspection.*—Inquiries emphasised the unsatisfactory position referred to in last year's Report in regard to the arrangements for the inspection of meat in certain districts, and instances are given showing that food inspection has apparently been discouraged by the local authorities.

3. *Bacterial Food Poisoning.*—Several outbreaks of this kind of poisoning were reported during the year, infected pork being the cause in most cases. In the borough of Chorley 131 cases of poisoning (one fatal) occurred after the consumption of pork pies and pork. Another outbreak at Openshaw, Manchester, resulted in three

deaths out of 163 persons affected; examination of the meat (pig's belly, pig's feet, pig's cheek, etc.) suspected of being the cause of the poisoning yielded unsatisfactory results owing mainly to the fact that only a small portion of the meat was available. Analysis of the blood and fæces from the patients and of post-mortem specimens from the deceased persons showed, however, that the outbreak was due to bacilli of the Gaertner group, probably derived from the meat consumed. A Report on Bacterial Food Poisoning and Food Infections has been issued during the year. (See ANALYST, 1913, **38**, 535.)

4. *Work in Relation to the Sale of Food and Drugs Acts.*—Investigations into local methods of administration of these Acts were made in a number of districts. The Kent County Council has under consideration a scheme under which it is proposed to place the control of the administration of the Acts in the hands of the county medical officer of health, to appoint a whole-time public analyst and assistants, to equip a county laboratory, and to appoint specially qualified sampling officers. Reports received from the borough of Kendal showed that, whilst no samples were submitted to the public analyst during the first and second quarters of 1912, a considerable number of samples had been sent for examination elsewhere. The Board notified the town council that they have no legal authority to pay for analyses of samples taken under the Food and Drugs Acts when such analyses were not made by the public analyst.

With the object of preventing the sale of adulterated milk as separated milk, the Board of Agriculture and Fisheries have issued Regulations which now require skimmed milk to contain at least 8·7 per cent. of solids-not-fat.

Unsuccessful proceedings were instituted by the Lanarkshire County Council in respect of a sample of flour reported by the public analyst to have been bleached with nitrogen peroxide; in giving judgment, the sheriff stated that, however desirable it might be to prohibit processes of food preparation which might in certain cases produce noxious results, such operations did not necessarily involve an offence against the Sale of Food and Drugs Acts. The County Council entered an appeal, but withdrew it on receiving advice on the sheriff's finding on the facts.

The practice of adding so-called "improvers" to flour appears to be a rapidly growing one, and is viewed with disapproval by the Board. The objection to such additions is especially emphasised in the case of flour, a substance on which the bulk of the nation depends to a large extent for its nutrition. The relation which may exist between apparently very minute alterations in the nature of staple food materials and the production of great and far-reaching changes in nutrition has been strikingly demonstrated in recent investigations into certain obscure disorders of metabolism, of which the disease known as "beri-beri" may be taken as an example. The time has arrived for taking a wider view of the danger to health arising from the sophistication of food-stuffs, and the Board are taking steps to promote legislation on the subject.

The Public Health (Milk and Cream) Regulations, 1912, are published as an appendix to the Report.

W. P. S.

INSTITUTE OF CHEMISTRY.

The Thirty-sixth Annual General Meeting of the Institute of Chemistry was held at 30, Bloomsbury Square, on Monday, March 2, Professor Raphael Meldola, President, in the chair.

After the routine business had been transacted, the President delivered his address. He referred to the progress of the fund for the new buildings of the Institute, and acknowledged the generous support of many companies, firms and individuals, other than members, who had contributed. Taking into account the promise of an anonymous benefactor to double every subscription received beyond £12,000 up to the amount of £2,500, it was probable that less than £1,000 was now required, but if more were received the Buildings Committee could easily invest it in improved and more serviceable finishings.

Referring to the endeavours of the Institute to secure fuller recognition for the profession of chemistry, the President recalled the evidence given by Sir William Tilden and Sir William Ramsay, as representatives of the Institute, before the Royal Commission on the Civil Service. The main portion of their evidence related to the conditions of service of chemists engaged in the Department of the Chief Inspector at Woolwich Arsenal. The Council of the Institute, in the memorandum submitted to the Royal Commission, stated that the chemical staff in that department should be controlled by a chemist of the highest efficiency. The rapid development of science in every direction was leading to increased specialisation. The real expert whose knowledge and experience were of most value to the community was the highly trained man who had specialised in some particular field. Surely such a man was the most competent to control the work of any public department which was concerned with his own subject. The training and experience which had raised him to his position of efficiency were no less protracted and severe than those required for the attainment of similar status in any other profession. Why, therefore, should there be this tendency to subordinate expert scientific service to non-expert control? The question of national defence was an important one, and the valuation and control of materials required in our arsenals, and the study and application of new discoveries having any bearing upon "the arts of war," were no less matters for chemical control than the conduct of an army in the field would be a matter for control by the General Staff. Yet, while the medical service took army "rank," the chemist, whose services were of equal importance, not only took no "rank" at all, but was made responsible to superiors having no special knowledge of his subject. This state of affairs, rendering as it did the public service of chemists an unattractive career to the best talent in the profession, was fraught with danger to the future well-being of the country, and was a short-sighted policy which, in time of trouble, might well lead to disaster. It was a matter which could not be lightly dismissed because it affected only a small number of chemists—it was a question of principle of far-reaching consequences, and it was to be hoped that the Royal Commission would give heed to the representations of the Institute.

Another move in this same direction was the rearrangement of the Chemical Staff of the London County Council which had been carried out since the retirement of Dr. Clowes. Under the new scheme the Chemical Department—as an independent department, ceased to exist, and the Chemical Staff was subordinated to the Medical Officer of Health. This, again, appeared to be a distinctly retrograde step, and one which the Institute could not but deplore. The matter had been represented to the Chairman of the County Council, Mr. Cyril Cobb, and he had made it perfectly clear that in this rearrangement there had been no intentional slur cast upon the status of the Chemical Staff; but it was a dangerous precedent, and one which the Institute regarded with apprehension for several reasons. There was the impression left on the public mind that the services of the chemist were of less importance than formerly, whereas in fact as time went on they were certain to become more and more important. Further, there was conveyed the idea that the status of the professional chemist was an inferior one; in other words, that his profession was to be degraded in rank, with corresponding exaltation of a kindred profession—a principle to which the Institute could not give its sanction.

The Council had also dealt with the conditions of appointments of Public Analysts, on which subject they had prepared a statement which had been published in the "Proceedings," and would be issued to members of local authorities. The Council hoped that by this publication the authorities might realise and appreciate more fully than they appeared to have done in the past the nature and responsibilities of these public officers.

In other branches of the profession, particularly in its applications to industry, the prospect for young chemists was improving. Not only was there little difficulty in placing Associates in appointments, but they were offered higher commencing salaries than formerly, and although the cost of living had greatly increased, they were generally able to secure a "living wage" in most branches of the profession at the outset of their careers, which was much less frequently the case a few years ago.

The President then dealt at considerable length with the Report of the Conference of Professors of Chemistry held under the auspices of the Institute in October last, which was attended by professors from practically all the principal educational centres of the country, the Institute thus providing an arena for the free discussion of the broad question of the education of professional chemists. The Report of this important meeting was under the consideration of a special committee, composed of representatives of every department of the profession—professors and teachers, private consultants, practitioners, and those who are accustomed to the scientific control of industries. Their task would be one of no little magnitude, and would probably effect considerable recasting of the regulations of the Institute in the light of modern educational development. Whatever scheme might eventually be adopted, it was certain that the Council would safeguard the status of the existing members. Professors and teachers would gladly welcome the support which the Institute might lend to their endeavours to raise the educational level to the required standard, while senates, councils, and governing bodies could not afford, on public grounds, to set aside curricula of studies, or to ignore a standard, regarded as essential for efficient training for the chemical profession by a body fully representative of that profession. It was in this way that the influence of the Institute in the educational world might, through a revision of the existing regulations, be exalted into a still greater power, making for the highest efficiency of the chemist of the future.

The following were elected Censors: Dr. George Beilby, F.R.S.; Professor Percy F. Frankland, F.R.S.; Mr. David Howard; and Dr. George McGowan.

The Officers and Council for the ensuing year were elected as follows:

President.—Raphael Meldola, D.Sc., LL.D., F.R.S.

Vice-Presidents.—George Thomas Beilby, LL.D., F.R.S.; Edward John Bevan; James Johnston Dobbie, LL.D., D.Sc., F.R.S.; Sir Alexander Pedler, C.I.E., F.R.S.; Sir Boverton Redwood, Bart., D.Sc.; Edward William Voelcker, A.R.S.M.

Hon. Treasurer.—Alfred Gordon Salamon, A.R.S.M.

Members of Council.—Leonard Archbutt; Robert Frederick Blake; Arthur George Bloxam; William Thomas Burgess; Cecil Howard Cribb, B.Sc.; Charles Frederick Cross, B.Sc.; Martin Onslow Forster, D.Sc., F.R.S.; Gilbert John Fowler, D.Sc.; Sir Richard Garton; Arthur Harden, D.Sc., Ph.D., F.R.S.; Otto Hehner; Charles Alexander Hill, B.Sc.; Edward Hinks, B.Sc.; William Richard Eaton Hodgkinson, Ph.D.; Alfred Henry Knight; Henry Rondel Le Sueur, D.Sc.; William Macnab; Gordon Wickham Monier-Williams, M.A., Ph.D.; Francis Richard O'Shaughnessy, A.R.C.S.; William Henry Perkin, LL.D., Ph.D., F.R.S.; Sylvester Oliffe Richmond; Clarence Arthur Seyler, B.Sc.; Thomas Stenhouse, Jr., B.Sc., A.R.S.M.; Frederick Wallis Stoddart; Oliver Trigger; William Henry Willcox, M.D., B.Sc.; James Woodward, B.A., B.Sc.

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REVIEWS.

THE MANUFACTURE OF SULPHURIC ACID AND ALKALI, WITH THE COLLATERAL BRANCHES. A Theoretical and Practical Treatise. By GEORGE LUNGE, Ph.D. Vol. I., Sulphuric Acid (in three parts). Fourth edition. London: Gurney and Jackson. 1913. Price £3 3s.

The first edition of this book was well called a "monumental" work. In that the portion devoted to sulphuric acid took up 650 pages. In the present edition the 650 have swollen to 1,600, in spite of excisions which make the earlier editions still necessary for consultation from a historical point of view; and the so-called "volume" consists of three bulky tomes. Nothing could better illustrate the enormous development of chemical industry during the past thirty years; and nothing could better strike terror into the heart of the would-be reviewer. But the work is so eminently a classic: it has for years been so well known: its author's qualifications as works manager, as investigator, as founder of a great school of chemical technology, and as a tireless worker, are so outstanding, that there is little for a reviewer to do beyond comparing the present volumes with the last edition, and noting the chief topics on which new matter has been added.

It should be said at once, however, that the encyclopædic character of the work has been fully preserved; not only have we matter new because its subject is new, but accounts of, and references innumerable to, new work on old subjects. Indeed, one of the merits of the book is the fulness of reference to originals, whether books, journals, patents, or personal communications. In Part I. we have a condensed but very clear account of the Frasch process, and a survey of numerous improvements in the methods of extracting sulphur from the native ore, and processes for obtaining it from sulphides. A good deal of space is devoted to nitric acid condensation, and Skoglund's and other apparatus are described, which yield a pure nitric acid by a single operation. Naturally, too, the processes for obtaining this acid from the atmosphere and from ammonia demand attention, and an admirable review of the whole subject occupies some fifty pages of the book. Knietsch's diagram of the physical properties of sulphuric acid of varying strengths is introduced in the chapter on the characters of the acid, and in the analytical section the analysis of fuming sulphuric acid and the detection of arsenic are naturally treated more fully than before. In the technical chapter on production of sulphur dioxide there are full accounts of the most recent form of the Herreshoff burner, of the Kaufmann and O'Brien burners, and of the enormous mechanical furnace of Wedge. The section on dust chambers and other appliances for cleaning the gas as it leaves the burners is also very considerably extended.

Part II. deals with the chamber process, from the Glover tower to the Gay Lussac tower, and with the purification of the chamber acid. Much of this volume—details of construction of chambers, etc.—is necessarily the same as in former editions; the new matter refers chiefly to the various devices which have been proposed of late years to effect so-called "intensive" working of the process—that is to say, to increase the yield per cubic foot of chamber space. We have, then, very full accounts of new forms of chambers, such as Falding's very high chambers and Meyer's tangential chambers, substitutes for or auxiliaries to chambers such as "reaction-towers," and means for urging the gases through the chambers and facilitating mixture—fans, water-sprays, etc. Connected with this are recent proposed modifications in packing for Glover towers, and in function of both Glover and Gay Lussac. There is a thorough compendium of all the voluminous literature on these subjects of the past ten years or so. Following this we have the working of the process and its theory; the space devoted to the latter greatly extended now, for there is a good account of the views of Raschig (with the author's views upon them), Wentzki, Jurisch, Divers, Bredig, Trautz, and others who have contributed so largely of late years to this increasingly complex and still controversial subject.

Part III. deals with the concentration of the acid, in which Kessler's hot-air method, Hartmann and Benker's cascade method, and the use of silica dishes, are all described; with the arrangement of works, and the question of yields and costs; and with the manufacture of fuming acid and sulphuric anhydride. This section naturally contains a great deal of new matter; that portion of it treating of contact processes runs to nearly 200 pages, and both the theoretical and the technological sides of the question are entered into with the author's characteristic thoroughness. The other chapters, on other sulphuric acid processes, on by-products, and on applications of the acid, differ but slightly from former editions, and the statistics are brought as nearly as possible up to date. Thirty-five pages of "addenda," matter published during the printing of the volume, give a further indication of the rapidity with which new work accumulates, and also of the vigilance of the author. On contemplating the book, and on reading the terse and vigorous preface, one gets strongly the impression of strenuous youth; yet facing the present reviewer is a presentment in bronze of the author, recording the fact that he celebrated his seventieth birthday five years ago. J. T. Dunn.

The Silicates in Chemistry and Commerce, including the Exposition of a Hexite and Pentite Theory of a Stereo-Chemical Theory of General Application. By Dr. W. Asch and Dr. D. Asch. Translated, with Critical Notes and some Additions, by Alfred B. Searle. London: Constable and Co., Ltd. 1913. Pp. 456 + xx. Price 21s. net.

The title of this book is somewhat misleading. The reader who expects to find in it a treatise on the chemical and commercial aspects of silicates will be disappointed; for the work does not merely include, but practically consists of, an exposition of the theories mentioned in the subtitle.

In their "Hexite-Pentite" theory the authors have endeavoured to explain the constitution of silicates stereo-chemically by the assumption of a ring-configuration built up of rings which are composed of six or five $Si(OH)_4$ or $Al(OH)_3$ molecules respectively. Although the authors draw upon the results of the majority of workers in the chemistry of clays and silicates generally for the support of their theory, the experimental evidence is hardly convincing enough to divest it of its highly speculative character.

The authors have utilised and digested an enormous material in order to prove the applicability of their theory to the constitution of natural silicates, clays, ultramarines, cements, glasses, and glazes, and to an explanation of crystalline structure; but it is highly doubtful whether their ambitious scheme, to bring the "Hexite-Pentite" theory into line with practically all achievements of physical and general chemistry (embracing hypotheses so far apart as Newton's law of gravitation and Ehrlich's side-chain theory), will be found in practice to be the "touchstone" of the structure of matter which the authors appear to see in it.

Whatever its practical value, the work is certainly interesting, and the translator is to be commended for undertaking the laborious task of rendering into English and providing with occasional notes a book which, after all, can only find a comparatively limited circle of readers. R. LESSING.

MAY, 1914. Vol. XXXIX., No. 458.

THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

An ordinary meeting of the Society was held on Wednesday evening, April 1, in the Chemical Society's Rooms, Burlington House. The President, Mr. A. Chaston Chapman, F.I.C., occupied the chair.

The minutes of the previous ordinary meeting were read and confirmed.

Certificates of proposal for election to membership in favour of Messrs. L. H. D. Acland, B.A., W. A. Gibbings, F. H. Lees, F.I.C., and W. H. Woodcock were read for the second time.

Messrs. R. Bickerstaffe, T. H. Byrom, F.I.C., S. G. Clifford, D. R. Frazer, A.I.C., and J. McLaren were elected members of the Society.

The following papers were read: "The Water of Dorton Spa," by C. A. Mitchell, M.A., F.I.C.; "On Damage caused to Vegetation by Sulphurous and Sulphuric Acids in the Atmosphere," by R. R. Tatlock, F.I.C., and R. T. Thomson, F.I.C.; and "Abnormal Refraction of Milk Serum," by J. McCrae, Ph.D., F.I.C.

ON DAMAGE CAUSED TO VEGETATION BY SULPHUROUS AND SULPHURIC ACIDS IN THE ATMOSPHERE.

By R. R. TATLOCK and R. T. THOMSON.

(*Read at the Meeting, April* 1, 1914.)

The damage to plants caused by sulphurous and sulphuric acids in the atmosphere has been the subject of a considerable amount of investigation, and the results of experiments are on record, showing the proportions of that gas in the air which would cause damage or death to plants. So far as our examination of the literature of the subject goes, we have not found any safe guidance as to the data obtained by analysis which would indicate whether a plant had been even probably damaged or destroyed by sulphurous or sulphuric acid (see Schlick's "Manual of Forestry," vol. iv.). It has long been recognised that sulphurous acid and sulphites absorbed by the leaves of plants are quickly oxidised into sulphates, and in our own experience no sulphite is ever present in plants, so that sulphates only require to be determined in order to find whether the proportion of these exceeds the normal, if, indeed, a normal quantity can be fixed. It has been proposed, and the plan has been adopted, to regard the fact of any decaying plant containing sulphates beyond certain limits as sufficient to account for such damage, but we propose to show that this is a very dangerous and unwarranted attitude to assume, and that it is by no means borne out by facts.

In determining sulphates in the leaves or other parts of plants, we find that they are all extracted by digesting 5 grms. of the somewhat coarsely cut up sample in a hot mixture of 100 c.c. water and 20 c.c. hydrochloric acid for several hours, or until the tissue is fairly well disintegrated, then filtering and precipitating with barium chloride in the usual way. As the organic matter appears to retard the precipitation of the barium sulphate, and is also apt to carry away with it some of that compound through the filter, if the latter is washed with hot water, we would recommend that the mixture should be allowed to stand for several hours, and that the precipitate should be washed with cold water.

We shall give in the accompanying table a series of results for sulphates (stated as sulphuric anhydride) in grass, bracken, and the leaves of various trees, as well as the soil in which some of these were grown, taken in different localities, after which we shall give explanations of these, and their bearing on the question of damage. The results given are calculated on the samples dried at 100° C., but the water originally present is also stated, so that the percentages of the other ingredients can be obtained, if required, in the samples as taken from the plant.

In the accompanying table of results the letter H means that the plants or trees from which the leaves, fronds, or needles, were taken were in a perfectly healthy condition, whereas those marked D were apparently quite damaged and decayed.

It will be necessary to explain shortly the general character of the localities from which the various samples were taken, so that the results obtained will be understood. The first is a village near the Gareloch, named Rosneath, and from the land around it the samples were obtained. This district is very sparsely populated, and the atmosphere is, therefore, pure and almost free from smoke. The samples taken in this locality may be regarded as practically free from pollution with sulphur products. Shettleston is now a portion of the City of Glasgow, and adjacent to it are many works using a large amount of coal, so that, although the atmosphere is not at its worst, it is still very considerably contaminated with the gases and smoke from the burning of coal and gas. Coatbridge, which is a town about eight miles from Glasgow, is in the centre of the iron industry, and there is probably more contamination in this district with coal smoke than in any part of Scotland. As regards the West Highlands, we are not in a position to state definitely the exact spot, but we may say that it is where very pure air and where air charged to a comparatively considerable extent with sulphurous or sulphuric acid may be found within a short radius, and this explanation will, no doubt, be sufficient to explain the results of our analysis. Langside is a portion of Glasgow, and, although not in the smokiest part of the city, it may be regarded as a sort of average between the worst and the best parts as regards pollution with smoke. The Midlothian samples were taken in a district where the probability of contamination was strong, and the results are conclusive as to this having taken place. The Ardrossan samples were taken rather less than half a mile from that seaport, and there would certainly be some contamination from the smoke from works, houses, and locomotives, probably also a little from steamers when the wind was blowing from the sea.

Before referring to the results of analysis, we should mention that, according to our experience, there ought to be two grounds on which opinion must be based before it can be said definitely that damage by sulphuric acid has taken place. These are—(1) The percentage of SO_3 found must be considerably above that present in the plant when grown in a fairly pure atmosphere; and (2) the leaf or other part of the plant when moistened on the surface with water ought to show an acid reaction with delicate blue litmus-paper. Most plants, and all those given in the table, when beaten with water and disintegrated, give a faint acid or amphoteric reaction; but some are fairly acid, such as rhubarb, and at least some varieties of begonia. When tested as described, without breaking the surface of the leaf, for example, none of the plants mentioned in the table give an acid reaction to litmus, but phenolphthaleïn is absolutely useless for this purpose. The importance of these conclusions will be apparent by a consideration of the analyses of the various plants, and we shall take them in the order given, beginning with grass. The results for SO_3 in three different districts are very instructive, as the amount of that ingredient is more than two and a half and three and a half times respectively in the smoky districts than in the purer atmosphere. In spite of these differences, all three were perfectly healthy and excellent grass, and none of them gave the acid reaction with litmus. Other samples (and this is also the case with all the plants) gave similar results; and although a maximum of 0·3 per cent. of SO_3 may be accepted as being present in grass grown in a fairly pure atmosphere, yet it may be perfectly good grass even if 1·0 per cent. is found, provided that it is not actually acid.

In the case of bracken, the healthy variety did not show acidity, but the damaged or decayed ones did; and yet in the latter there was only 0·356 per cent. of SO_3, but still more than double that found in the undamaged plant. We may mention that we have tested bracken that decayed naturally, but no acidity was found.

In the case of the oak leaves, we have illustrated in an absolutely complete manner the data we have stated as being necessary to come to a definite conclusion. The leaves from Rosneath, one from the West Highlands, and that from Langside, did not show acidity, although the last-named contained up to 0·420 per cent. of SO_3. The other sample from the West Highlands was acid and damaged, and contained only 0·202 per cent. of SO_3; but both of the Midlothian samples were acid and damaged, one of them containing little more SO_3 than the undamaged and healthy Langside oak leaves. It would appear to be conclusively proved from these results that the percentage of SO_3 in a plant is no criterion as to whether it has been actually damaged by acid, but that it is in addition necessary to prove actual acidity.

The results recorded for birch leaves are also very instructive, as healthy and neutral leaves from Langside contained 0·86 per cent. of SO_3, whereas damaged and acid leaves from the West Highland locality only contained 0·248 per cent.

The results for hawthorn leaves, rhododendron petals and leaves, and sycamore leaves, all go to show that mere excess of SO_3 over the normal is no proof of damage, but that presence of acidity is an essential factor. The "new" leaves of the rhododendron are those which grew in the year mentioned, and the "old" leaves were those which were still in a healthy condition on the plant, but had grown in previous years.

It might be argued that the excess of SO_3 in some of the plants was due to the presence of sulphates in the soil, but from the analyses given in the table it is evident that there is little or no ground for such a contention, except to some extent in the Coatbridge example. It is much more probable, however, that the excess over the normal in the Langside, Shettleston, and Coatbridge samples is due mainly to absorption of sulphate of ammonia from the atmosphere. It will be understood that although we hold that it is necessary to find both acidity and excess of SO_3 in order to *prove* damage by acid, yet it must be admitted that the latter item may point to the possibility of such damage having been caused. For example, acid may actually have attacked vegetation, and have been subsequently washed away by rain. In such circumstances the leaves or other part of the plant should be tested occasionally just where they are growing, and especially during dry weather. These tests should also be made at various parts of a plantation of trees, for example, as the damage may be local to a large extent, and particularly in the line of the prevailing wind. In these ways we have been successful in finding acidity, by litmus as indicator, where it was really present; but even in smoky parts of Glasgow we have never detected acid on the leaves of plants, nor by bubbling large volumes of air through water. We would here put in a word of caution as to the use of phenolphthaleïn, by which we are convinced many fallacious results have been recorded, as it has been adopted for similar purposes to the present, evidently without proper thought having been given to its real functions and limitations. Thus, in testing the leaves of plants or air with that indicator, we may be actually recording normal salts of ammonia or other base as if they contained free acids. In addition to the tests referred to, we have also collected rain in various localities, smoky and otherwise, but the water has never been acid, but always faintly alkaline. This result is undoubtedly brought about by the sulphuric acid derived from the coal having been neutralised by ammonia also produced from the coal, by ammonia which is invariably present in the atmosphere, and by bases, such as lime, which are present in the floating dust in the air. From all our observations we have come to the conclusion that the atmosphere, even of cities consuming large quantities of coal, is only acid under very exceptional circumstances, and even then within narrow limits. For example, acidity has been detected where acid gases from a works, or from the burning of coal containing excessive sulphur (in rare instances 5 to 6 per cent. has been found by us), find their way into the air. In such cases damage to vegetation and corrosion of ironwork has been observed by us; but the effect has been strictly local, and when the acid has become dispersed through the large volume of surrounding air no damage appeared to be caused and no acidity could be detected.

We may just add that we have had on hand trials in order to determine to what extent SO_3 enters into the composition of plants, if the soil in which they are grown is kept impregnated with sulphates, such as those of calcium or magnesium; but as our results are not completed we may make them the subject of another communication.

Plant.	Locality.	Date.	Healthy or Damaged.	Per Cent. in Sample dried at 100° C.		Per Cent. in Original Sample.
				SO_3.	Mineral Matter.	Water.
Rhododendron, red petals	Rosneath	May 31, 1912	H.	0·117	3·32	88·06
Rhododendron, lilac petals	Rosneath	May 31, 1912	H.	0·133	2·86	89·48
Rhododendron, lilac petals	Langside	May 28, 1912	H.	0·330	4·29	89·71
Rhododendron, new leaves	Rosneath	May 31, 1912	H.	0·116	3·86	78·40
Rhododendron, new leaves	Langside	May 28, 1912	H.	0·332	5·21	75·35
Rhododendron, old leaves	Rosneath	May 31, 1912	H.	0·120	3·97	56·14
Rhododendron, old leaves	Langside	May 28, 1912	H.	0·496	4·12	51·66
Sycamore leaves ..	Ardrossan	August 3, 1913	H.	0·342	8·10	78·10
Sycamore leaves ...	Langside	August 6, 1913	H.	0·629	10·86	76·00
Larch leaves ...	Rosneath	June, 1912	H.	0·190	3·31	66·24
Scots pine (or fir) needles	Rosneath	June, 1912	H.	0·157	3·03	65·72
Spruce fir needles...	Rosneath	June, 1912	H.	0·145	2·85	62·91
Wellingtonia gigantica needles	Rosneath	June, 1912	H.	0·191	4·47	65·58
Soil...	Rosneath	June, 1912	—	0·027	—	—
Soil...	Langside	August, 1913	—	0·038	—	—
Soil...	Ardrossan	August, 1913	—	0·035	—	—
Soil...	Coatbridge	August, 1911	—	0·083	—	—
Grass	Rosneath	June, 1911	H.	0·271	7·43	75·25
Grass	Shettleston	July, 1911	H.	0·720	8·32	71·40
Grass	Coatbridge	August, 1911	H.	0·987	10·61	77·11
Bracken	West Highlands	July, 1912	H.	0·159	6·41	78·63
Bracken	West Highlands	July, 1912	D.	0·356	7·07	76·68
Oak leaves... ...	Rosneath	June, 1912	H.	0·147	4·32	67·39
Oak leaves... ...	West Highlands	July, 1912	H.	0·109	4·67	65·32
Oak leaves... ...	West Highlands	July, 1912	D.	0·202	5·47	69·88
Oak leaves... ...	Langside	June, 1912	H.	0·420	4·73	65·76
Oak leaves... ...	Midlothian	September, 1911	D.	0·476	6·87	61·14
Oak leaves... ...	Midlothian	September, 1911	D.	1·385	7·73	54·37
Birch leaves ...	Rosneath	July, 1912	H.	0·127	0·407	67·62
Birch leaves ...	West Highlands	July, 1912	D.	0·248	3·75	58·46
Birch leaves ...	Langside	June, 1912	H.	0·860	3·32	56·43
Hawthorn leaves ...	Rosneath	June, 1911	H.	0·154	7·08	60·59
Hawthorn leaves ...	Coatbridge	August, 1911	H.	0·653	6·16	60·10

DISCUSSION.

The **PRESIDENT** having invited discussion,

Dr. **DYER** said that the subject of damage to trees had been investigated many years ago by the late Professor Stöckhardt.* It was obvious that, as the present authors stated, the mere presence of sulphates was not evidence of external contamination, unless there was also acidity. Lately he had met with an interesting instance of the effect of sulphurous acid, not on leaves, but on fruit—oranges and lemons. A consignment of these had been unpacked in a damaged condition from a ship which had been fumigated with sulphurous acid, presumably for the destruction of vermin. The oranges and lemons were flaccid, and in some cases shrivelled, and their surfaces gave evidence of the presence of traces of sulphuric acid. He found by experiment that when sound oranges and lemons were kept for twenty-four hours in an atmosphere of sulphurous acid they shortly afterwards became soft, and gradually shrivelled. Apparently the effect of the sulphurous acid was to kill the external cells of the skin, facilitating transpiration of moisture.

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* See *Biedermann's Centralblatt für Agricultur-Chemie*, vol. i., p. 108, and vol. v., p. 362; also *Der chemische Ackermann* for 1872, pp. 24-27 and 111-121. See also papers by J. Konig in *Biedermann's*

Mr. W. PARTRIDGE questioned the suitability of litmus for testing acidity in a case like this, and suggested that probably Congo red or tropæolin would give better and more differential results.

Mr. E. M. HAWKINS remarked that metanyl yellow was a most delicate indicator for mineral acids, while it was not affected by sulphurous or nitric acid.

Mr. A. R. TANKARD said that damage to a plant might arise from a number of causes, of which the taking up of sulphurous acid was only one. In the case particularly of leaves, for instance, damage probably occurred before there was any great concentration of the acid in the living vegetation, and it had been shown by Cohen, and by Crowther at Leeds, and others, that the stomata of leaves became blocked up, causing the death of, or serious damage to, the plants; and this often occurred long before any serious amount of contamination from sulphurous or sulphuric acid was detectable by chemical methods.

Dr. KEANE said that the authors' chief object was presumably to point out that too much reliance should not be placed upon determinations of the total sulphuric acid in air, and that due weight should be given to the quantity of free acid present. Several years ago some experiments on the same subject were made at Kew by Sir W. T. Thistleton Dyer; and a number of chemists, with whom he (the speaker) was associated, had arranged, under the direction of Dr. G. H. Bailey, a plan for analysing the air of towns, stations being established for the estimation of SO_2 in air, rain, and snow, and for investigating the effects of SO_2 on plants. Such stations were established in Manchester, London, and Liverpool, and the results of the work were contained in various papers published by the Manchester Field Naturalists' Society, Dr. Russell, Dr. Oliver, and others, between 1891 and 1895.

From the data given by the present authors it was a little difficult to see whether their experiments were made under conditions that were normal, or nearly so; whether, for instance, the three samples of grass referred to were of such a character as might be expected in a healthy atmosphere, and whether, even if the grass itself was healthy, the quantity produced per acre was not less than would be the case in the absence of any poisonous emanations. Those who had seen the deserts which surrounded Widnes and St. Helens would have a very practical idea

Centralblatt for 1878, 1879 and 1881, and by J. Schröder in the *Versuchsstationen Organ*, vol. xv., pp. 321-355. Abstracts of the last-named paper and of Professor Stöckhardt's paper in *Der chemische Ackermann* may be found in the *Journal of the Chemical Society* for 1872, vol. xxv.

of the effects which such gases could produce. The results obtained by chemical examination were effected to a considerable extent by the atmospheric conditions. While in clear weather in, say, May or June, but a few tenths of a grain of SO_2 per 100 cubic feet might be found in the air; in November or December there might be from 2 to 4 grains per 100 cubic feet, and the effect of fog would be to increase this quantity to from 10 to 20 grains per 100 cubic feet. In Manchester the quantity was far in excess of that found in Liverpool; while in London, near St. Bartholomew's Hospital, the late Dr. Russell had found an extraordinary aggregation of sulphur compounds, with marked acidity, amounting to between 30 and 38 grains of SO_2 per 100 cubic feet during a dense fog in December. The effect upon plants was a cumulative effect, which it was difficult to assign to any specific cause. For instance, the ammonium salts formed might produce a secondary clogging effect, apart from the distinctly poisonous effect of the sulphurous acid in the air. In connection, therefore, with investigations of this type, it was desirable to determine what quantities of SO_2 were normal to the air at the time of the investigation.

Dr. Voelcker said that it was very clear from the figures which had been given in the authors' paper that no dependence could be placed on the particular amount of sulphates found on leaves, etc., as a proof whether injury to vegetation had been done or not. For instance, it would be noted that with one kind of tree there had been destruction, though the figure recorded for the sulphates present was considerably lower than in the case of a different kind of tree which had suffered no damage.

Alike with the determination of sulphates and that of acidity in the case of leaves, grass, etc., much depended upon how the material was treated, whether merely the surface had been washed, or whether the material itself had been broken up.

Moreover, it was frequently the case that injury might have been done and yet the proof of it have been removed by subsequent washing by rain, etc. Where he had found such determinations to be of use was when it was possible to institute a comparison between herbage, trees, etc., placed in the direct line of fumes emanating, say, from a chimney or kiln, and similar equidistant but not thus subject, as determined by the prevailing wind. Different classes of trees differed much in regard to their relative susceptibility to the influence of noxious gases and fumes, and this needed to be taken into consideration also.

Mr. A. E. Parkes asked whether any injury had been found to result from the use of sulpur sprays for the destruction of insect pests.

Mr. J. Golding said that in hop growing sulphur was constantly used for the purpose of getting rid of mould, and he remembered an apparatus in which the sulphur was actually burnt; but as far as he knew, there was no appreciable damage done to the hops.

Dr. Voelcker said that he had, at the Woburn Experimental Farm, tried the application of sulphur to the soil, but had so far failed to find any benefit produced; and, indeed, in one case where sulphur was added to the extent of its forming 0·4 per cent. of the soil, the effects with mangold appeared to be even hurtful.

Mr. G. Embrey said he thought that when the leaves of the damaged plants, on being washed with water, yielded distinct traces of free sulphuric acid, this might almost invariably be taken as evidence that the damage was due to sulphurous acid fumes. In a case, many years ago, in which plants in a nursery garden had been damaged by emanations from a brick-kiln, he had had no difficulty in ascertaining the direction in which the fumes were passing, and in obtaining evidence of their effect on the plants.

Mr. C. A. MITCHELL said that the application of sulphur to the soil was said to be practised in the north of France for the purpose of increasing the crop, and it might be that the sulphur, while causing some injury to the plants themselves, simultaneously improved the condition of the soil by its action as a germicide.

Dr. LESSING said that about two years ago a committee was formed for the investigation of atmospheric pollution, and had succeeded in inducing a number of municipal and county authorities to set up an apparatus, devised by the committee, called a "soot-gauge," in which the rain-water was so collected that the carbon or soot and the tarry matters deposited from the atmosphere could be recovered. Monthly analyses were being made, and it was hoped that after a time the results would yield useful information. The question of the tarry matter was an important one in connection with the damage that was being done. All soot contained tarry matter, the quantity, according to his experience of a large number of samples, being rarely less than 5 per cent.

Mr. H. F. KNIGHT thought that some quantity of sulphurous acid might be present over and above that which acted poisonously on the plant. This seemed to be indicated by the differences shown in the case of the three samples of grass examined by the authors, and he suggested that the results would more closely represent the effect on the plant if the leaves were just rinsed before being tested.

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THE WATER OF DORTON SPA.

BY C. A. MITCHELL, B.A., F.I.C.

(*Read at the Meeting, April* 1, 1914.)

EARLY in the nineteenth century, at the close of the war with France, there was a sudden rush of visitors from this country to the spas on the Continent, and many of the old English watering-places were practically abandoned. Simultaneously with this neglect of our spas there began another movement of a largely speculative character, to exploit new spas which should rival in attractiveness those abroad. Among the most spirited of these attempts was the spa which was built about 1830 at Dorton, in Buckinghamshire. The spring thus developed into Dorton Spa had been known to the inhabitants of the district for many generations, and owing to its inky taste was commonly described as the "alum well." The ground for a radius of many feet was stained a deep yellowish-brown from the deposition of iron oxide and sulphate. According to a contemporary account, it was mainly used as a bath for mangey dogs and for "tanning" them for bull-baiting.

In a pamphlet, published in 1833 by the local surgeon, T. Knights, to advertise the spa, an analysis of the water made by Brande in 1830 is quoted. In spite of vigorous advertising the spa never became popular, and before the end of the forties the pump-room was pulled down and the building allowed to fall into ruins.

According to Brande, the water had a sp. gr. of 1·004 at 60° F., and was acid to litmus, this being due to the presence of 0·8 per cent. of free sulphuric acid.

It had the following composition in grains per pint:

Sulphate of lime	11·5
Muriate of soda	1·4
Sulphate of alumina	2·1
Sulphate of iron	10·0
Total solids	25·0

It contained also traces of carbon dioxide, nitrogen, and silica.

Last year I had the opportunity of examining the water of this well, which is undoubtedly of a remarkable character. The water had a sp. gr. of 1·0041 at 15·5° C., and on evaporation at 100° C. left a residue of 433·2 parts per 100,000. This still contained water of hydration, which could be expelled at a higher temperature. Analysis of the salts present gave the following results, and for convenience of comparison I have calculated Brande's results upon the same basis of parts per 100,000:

	Mitchell (1913).	Brande (1830).
Iron	45·6	32·00
Aluminium ...		3·52
Calcium	40·0	31·30
Magnesium ...	15·0	—
Sodium	8·5	6·29
Sulphuric acid (SO_4)	243·0	202·89
Chlorine	10·0	9·71
	362·1	285·71

The difference between the sum of the constituents and that left on evaporation at 100° C. was due, as mentioned above, to water of hydration retained by the iron sulphate.

From a comparison of the analyses, it will be seen that the amounts of sodium, calcium, and chlorine, have remained fairly constant. Magnesium, if present eighty years ago, was overlooked by Brande. The great increase that has taken place in the total solids is due to the iron sulphate. A slight trace of aluminium is present, and this is included in the iron. As in the case of the earlier analysis, the water is acid, the acidity calculated as free sulphuric acid being 0·1 per cent.

DISCUSSION.

Dr. VOELCKER asked whether Mr. Mitchell knew what was the cause of the peculiar properties of this water, and whether there were any other waters of a similar nature in the district. He had had a good deal to do with water-supplies in Buckinghamshire, but had never met with this spring or any other of a like nature there.

Mr. T. Tickle asked whether the iron was wholly ferrous or partly ferric.

Dr. Lander asked whether Mr. Mitchell had made any observations as to whether the water had any radio-active properties.

Mr. P. A. E. Richards said that some years ago he had made an analysis of water from Boston Spa, near Harrogate (see Analyst, 1901, **26**, 68-71), which was found to contain barium. An analysis of water from the same source had been made in 1784, the results of which were fairly consistent with those of his own analysis, except that the barium was not discovered, but was included as calcium.

Mr. E. Hinks asked whether it was quite certain that the acid present was free sulphuric acid, and whether the water was free from carbonic acid.

The President remarked that presumably the free sulphuric acid would be due to some hydrolysis of the sulphate of iron.

Mr. C. A. Mitchell, in reply, said that he was not surprised that Dr. Voelcker did not know of the existence of this spring, since it was, as he had said, situated in a wood, and was difficult to find. He knew of nothing that would account for its peculiar properties. He had not ascertained in what state the iron was present, nor had he made any investigation as to the possible radio-activity of the water, though this seemed a not unlikely possibility, and might perhaps account for the striking effects with which the water had been credited. He had not determined whether the acid was actually sulphuric acid. No carbonates were included in the dissolved salts. He believed that in the earlier analysis the dissolved gases were found to include some carbon dioxide, but in his own case the quantity of water available was insufficient to enable this to be investigated. The water as it came from the spring was yellow and fluorescent from the particles of iron in suspension. These, however, soon subsided. After the water had been kept for about three months the deposit amounted to about 3·3 per cent. of the total solids. The deposition was slow, as the acidity tended to keep the iron in solution.

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ABNORMAL REFRACTION OF MILK SERUM.

By J. McCRAE, Ph.D., F.I.C.

(*Read at the Meeting, April* 1, 1914.)

The process for the detection of added water in milk by determining the refractive power of the serum has been advocated by Leach (in conjunction with Lythgoe), Mai, Rothenfusser, and others. I have already (*Annual Reports*, 1906, 1907, 1908) commented favourably on the process after extensive trials with the milk produced in the Transvaal. As a result of these tests it was found that, when the serum is obtained by coagulating the protein and fat by means of acetic acid (Woodman, *J. Amer. Chem. Soc.*, 1899, **21**, 503), the minimum refraction is 39 on the Zeiss immersion refractometer at 20° C. with unwatered milk; and when the coagulation is carried out by means of copper sulphate solution (*Annual Report for* 1908, *State Board of Health, Massachusetts*, p. 594) the minimum refraction is 36.

Two recent cases of abnormal milks have been investigated.

In the first case a sample of milk purchased under the local food by-laws gave the following results :

Fat	3·00 per cent.	
Non-fatty solids	7·06 "	
Ash	0·80 "	
Refraction at 20° C. of serum obtained by means of copper sulphate		34·4

These results pointed to the conclusion that water had been added to the milk. (It may be mentioned that, as the natural waters of this country are practically free from oxidised nitrogen, the detection of added water in milk by the identification of nitrates is impossible.) The vendor denied the addition of water, and sought to explain the anomaly of the results by stating that the milk was possibly derived from one cow only, and he was aware that this cow "was not giving good milk." To test this statement, the cow was milked at 4.30 p.m. in the presence of an inspector, under conditions which precluded all possibility of tampering, and this milk, after being efficiently mixed, was sampled and examined, with the following results :

Fat	2·70 per cent.	
Non-fatty solids	7·04 "	
Ash	0·82 "	
Refraction at 20° C. of serum obtained by means of copper sulphate solution		34·8

The cow was a Friesland, and had customarily been milked at 4.30 a.m. and 4.30 p.m.

The second case is of the same nature. The sample of milk purchased by the Food Inspector gave the following results :

Fat	3·00 per cent.	
Non-fatty solids	7·50 "	
Ash	0·77 "	
Refraction at 20° C. of serum ($CuSO_4$)		34·4

The vendor of the milk denied adulteration, and offered his herd for examination. The herd consisted of eighty Friesland cows, which at the time were being fed on mangels. Six of the cows were selected, and were milked at the usual evening milking-time in the presence of inspectors. The milk from these six cows was thoroughly mixed and sampled. The milk on examination gave the following results :

Fat	3·50 per cent.	
Non-fatty solids	7·83 "	
Ash	0·78 "	
Refraction at 20° C. of the serum obtained by means of copper sulphate solution		35·3
Refraction at 20° C. of the serum obtained by means of acetic acid		37·5

Seven days later the same cows were subjected to a second test, but on this occasion the milk from each cow was thoroughly mixed and sampled; then the milks from the six cows were mixed and sampled. The following results show that four of the cows were producing milk the serum of which possessed abnormally low refracting power:

	Fat per Cent.	Non-Fatty Solids per Cent.	Ash per Cent.	Refraction at 20° C. of Serum ($CuSO_4$).
Cow No. 1	2·7	6·20	0·77	32·3
„ No. 2	4·2	7·86	0·74	35·3
„ No. 3	3·7	9·05	0·74	37·4
„ No. 4	3·5	7·83	0·69	36·8
„ No. 5	2·5	7·15	0·69	35·8
„ No. 6	3·3	7·29	0·80	33·4
Mixed milk... ...	3·3	7·38	0·73	35·3
Average of 1 to 6 ...	3·3	7·56	0·74	35·2

All the cattle were apparently in good condition; they were well housed, and at the time of the tests the veld grass was plentiful.

I am indebted to Mr. H. J. Mann for the analytical results recorded here.

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DISCUSSION.

Dr. MONIER-WILLIAMS said that it would have been interesting to know what were the proportions of lactose and of chlorine in these cases. In a recent paper by L. Mathieu and L. Ferré (*Ann. Falsif.*, 7, 12, 1914), it had been asserted that if the chlorine was multiplied by an appropriate factor and added to the lactose a figure was obtained which was practically a constant, after correcting for the volume occupied by the precipitated proteins and fat—*i.e.*, the chlorine varied inversely as the lactose. This would be seen from the following results of some recent analyses which he had made of milk from individual cows, samples of which had been obtained for him by Mr. Golding:

Lactose.	Chlorine (as NaCl).
5·12	0·131
4·80	0·140
4·55	0·181
3·41	0·308
1·80	0·398
0·71	0·555

It was somewhat doubtful, however, whether the connection between lactose and chlorine was sufficiently close to be of any value in the analysis of mixed milks of normal character.

From the analyses given by the author it was possible that the lactose was abnormally low and the chlorine high. One would naturally expect that in such cases the refraction of the serum would be low, since the refraction was practically a measure of the lactose present, and was not affected to any great extent by the soluble salts. This relationship between the chlorine, as representing the soluble ash, and the lactose would appear to have been confirmed to some extent by L. C. Jackson and A. C. H. Rothera (*Biochem. J.*, 1914, **8**, 1). They showed that the conductivity of milk varied inversely as the lactose. The conductivity was practically a measure of the soluble salts, and was unaffected by the amount of lactose present.

It was probable that milk samples giving an abnormally low value for refraction would show a corresponding increase of electrical conductivity, and that a combination of the two values would give results comparable to those obtained by Mathieu and Ferré from a consideration of the lactose and chlorine present in the milk.

Mr. J. GOLDING said that the samples to which Dr. Monier-Williams had referred were very abnormal. The cows were drying off, but were in perfect health. One of them, just previously to drying off, gave milk that was in a clotted and stringy condition.

Mr. E. M. HAWKINS said that Dr. Monier-Williams's remarks reminded him of some samples of abnormal milk upon which Mr. Sidney Harvey read a paper before the Society some years ago. The samples came from the district round Canterbury, and the percentages of non-fatty solids were similar to those found in Dr. McCrae's samples, while the ash was very high. No explanation was then arrived at, but possibly the samples would have yielded results similar to those now obtained by Dr. Monier-Williams.

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

Poisonous Bran. Marchadier and Goujon. (*Ann. Falsif.*, 1914, **7**, 77-81.)—Bran which has been stored for some time undergoes decomposition, and becomes injurious when used as a food for animals, deaths having resulted from this cause. The decomposition proceeds most rapidly at the centre of a sack of bran, and, without going into the nature of the toxic substances produced, the author points out that the decomposition is accompanied by a decided increase in the acidity of bran, and that the acidity may be taken as a criterion of the wholesomeness or otherwise of this feeding-stuff. Normal bran has an acidity of less than 0·15 per cent. expressed in terms of H_2SO_4; an acidity of 0·15 to 0·30 per cent. indicates that decomposition has commenced, although the bran may not yet be injurious to health, whilst bran having an acidity of 0·30 per cent. or more should not be employed for feeding animals.

W. P. S.

Butter Fat Content of Margarine. W. Arnold. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1914, **27**, 379-388.)—The presence of butter fat in margarine may be ascertained by estimating the Reichert-Meissl, Polenske, and saponification values of the fat itself and of its alcohol-soluble portion, and by determining the molecular weight of the volatile fatty acids obtained in the Reichert-Meissl process, the whole of the results obtained being taken into consideration. In the first place, butter fat, coconut oil, and palm kernel oil, are not present in a margarine which yields Reichert-Meissl and Polenske values of less than 0·55. When the fat gives a Polenske value of not more than 0·6, or of 1·1 for the alcohol-soluble fat, and the molecular weight of the volatile fatty acids lies between 97 and 107, butter fat, but not palm oil, is present. The molecular weight of the volatile fatty acids rises to 130 to 144 when coconut oil is present, and the other values also increase. The relation between the Reichert-Meissl value and the Polenske value indicates whether coconut oil or palm kernel oil is present; the following table shows the relation in the case of margarines containing varying quantities of coconut oil:

	Coconut Oil per Cent.									
	2	6	10	14	20	25	30	35	40	50
Reichert-Meissl value	0·90	1·65	2·37	3·20	3·9	4·5	4·84	5·34	5·80	6·77
Polenske value ...	0·55	0·95	1·30	1·65	2·4	3·0	3·65	4·25	5·05	6·55

When the Reichert-Meissl value exceeds that given in the table, butter fat is also present; a lower value indicates the presence of palm kernel oil. The molecular weight of the volatile fatty acid lies between 130 and 136 when palm kernel oil alone is present in the margarine. W. P. S.

Detection of Beef Fat in Lard by Polenske's and Bömer's Methods. K. Fischer and J. Wewerinke. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1914, **27**, 361-378.)—The "difference value" method proposed by Polenske for the detection of beef fat in lard (ANALYST, 1907, **32**, 382; 1908, **33**, 476) does not always yield trustworthy results, and the authors, as the result of their experiments, are of opinion that a normal "difference value" (over 19) does not indicate definitely that a lard is genuine, and that a low value is not an absolute proof of the presence of beef-fat. Although Bömer's method (ANALYST, 1914, 84) is more rapid and convenient, it does not appear to be more sensitive than Polenske's method. W. P. S.

Skilfully Adulterated Cacao Butter. C. Grimme. (*Chem. Rev. Fett u. Harz-Ind*, 1914, **21**, 47-49; 74-75.)—A commercial brand of cacao butter, originating from Amsterdam, is sold under the name "Elite" (trade-mark, a swan) and is openly stated to be adulterated with other fats, but in such a way that detection is impossible. The price is from 5 to 10 per cent. lower than that of the pure product. Two samples examined by the author gave the following results: Sp. gr. at 65° C., 0·8800 and

0·8828; at 15° C., 0·9150 and 0·9178; melting-point, 30° C. and 30·4° C.; solidification-point, 28° and 28·1° C.; refractive index at 40° C., 1·4559 and 1·4558; acid value, 2·1 and 1·95; saponification value, 198·4 and 198·0; iodine value, 38·3 and 38·14; Reichert-Meissl value, 0·82 and 0·94; and Polenske value, 1·5 and 1·52. *Fatty Acids.*—Melting point, 46° to 48° and 46° to 47·5° C.; solidification-point, 45° and 44·9° C.; refractive index at 40° C., 1·4552 and 1·4554; neutralisation value, 207·0 and 206·3; iodine value, 38·82 and 38·53, and mean molecular weight, 271·0 and 272·1. The only notable differences between these results and those given by pure cacao butter were the lower melting-point of the fatty acids (which were also brownish-yellow instead of pale yellow or white) and the Polenske values. The critical temperature of solution in glacial acetic acid (35° C., as compared with 65° C. to 69° C. for genuine cacao butter) indicated the presence of a foreign glyceride. By fractional crystallisation of the fats from a mixture of anhydrous ether and alcohol (3 : 1) at 10° C., the adulterated fats yielded first fractions which were semi-solid and second fractions melting at 39·7° C. and 40° C., whilst four samples of pure cacao butter gave first fractions melting at 46° to 52° C., and second fractions melting at 56° to 57·4° C. The nature of the foreign ingredient was not identified.

C. A. M.

Microchemical Reaction of Caffeine, Theobromine, Theophylline, and their Derivatives, with Mercuric Chloride. M. Wagenaar. (*Pharm. Weekblad.*, 1914, **51**, 23-25; through *Chem. Zentralbl.*, 1914, I., 1026.)—The microchemical reaction with mercuric chloride is available for the recognition of purine derivatives with one or more methyl groups. In the case of caffeine and theophylline the precipitate consists of rosettes of crystals and spherolites; in the case of theobromine it consists of smaller masses of crystals. In this way 100 μ of caffeine, 100 μ of theophylline, and 50 μ of theobromine, can be identified easily. One μ of caffeine can be detected by drying the precipitate and breathing on it; 5 μ of theobromine by drying; 10 μ of theophylline react after some time. The reactions can be applied to various medical preparations containing salts or derivatives of these drugs; antipyrine-caffeine or "migranine" does not react. The test may also be used for the detection of the above purine derivatives in plants. J. F. B.

Flame Test for Chloral Hydrate. W. M. Doherty. (*J. and Proc. of Roy. Soc. of New South Wales*, 1913, **47**, 163.)—Chloroform when evaporated will colour the flame of a Bunsen burner in the vicinity intensely green, or if the quantity of chloroform is large, an electric blue. It is possible to detect extremely small quantities of chloroform in relatively large volumes of water by placing the mouth of the bottle containing it in juxtaposition with the air orifice of the burner which should have a well-cleaned "rose" top to it. An improvement of the test lies in the use of a type of spectrum burner designed for the detection of boric acid, a piece of clean copper wire being placed in such a position on the burner as to be outside the inner cone of the flame. If a solution containing 1 drop of chloroform in 100 c.c. of water after shaking well be placed in the glass arm attachment of the burner, a distinctly green flame is produced, any volatile halogen compound such as hydrochloric acid will give the colour, though it is not as active as chloroform. If a solution of chloral hydrate be placed in the glass portion of the spectrum burner, there is no coloration of the flame, but on adding caustic alkali an immediate coloration proportionate to the amount of chloral hydrate is produced, due to the liberation of chloroform.

H. F. E. H.

Estimation of Crude Fibre in Finely Powdered Substances. J. Grossfeld. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1914, **27**, 333.)—In the estimation of crude fibre in such materials as cocoa, flour, etc., by König's method (ANALYST, 1903, **28**, 318; 1908, **33**, 98), the glycerol-sulphuric acid mixture containing the crude fibre should be filtered without the aid of the suction-pump; if suction is applied, the fine particles tend to penetrate the filter to a certain extent, and the filter soon becomes clogged.

W. P. S.

Estimation of Ivory-Nut Meal in Flours and Fodders. J. Gerum. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1914, **27**, 392-394.)—Since it has been stated that ivory-nut meal (the ground offal obtained in the manufacture of buttons from the ivory-nut) is employed as an adulterant of flour, etc. (*cf.* ANALYST, 1907, **32**, 218), the author has devised the following method for the estimation of ivory-nut meal in mixtures of the same with various flours; the method depends on the fact that ivory-nut meal has a sp. gr. exceeding that of chloroform. Five grms. of the flour are shaken with chloroform, and the mixture is allowed to settle for six hours; the sediment is then collected on a filter, washed with chloroform, dried at 105° C., and weighed. Ivory-nut meal contains on an average 10 per cent. of water, and the weight of the sediment is corrected by this amount in order to obtain the quantity of the meal present. The amount of ivory-nut constituents soluble in chloroform may be neglected, since it only amounts to from 0·6 to 1 per cent. of the substance. In four experiments with ivory-nut meal itself, the quantities recovered (corrected for moisture) varied from 95·9 to 100·5 per cent. of the quantity taken. Three wheat-flour mixtures, containing 20, 25 and 50 per cent. of ivory-nut meal, yielded 18·8, 22·7, and 47·1 per cent., respectively. Other figures are recorded showing that the method gives approximately correct results. The sediment obtained should always be examined microscopically. Two samples of wheat and rye meals, which had been unsuitably stored for two years and had undergone decomposition, yielded considerable quantities of sediment when treated with chloroform; these sediments were soluble in 3 per cent. sodium hydroxide solution, and a microscopical examination showed that ivory-nut meal was not present.

W. P. S.

Schardinger's Formaldehyde-Methylene Blue Reaction and Other Ferment Reactions of Goat's Milk. W. Wedemann. (*Biochem. Zeitsch.*, 1914, **60**, 330-344.)—Goat's milk, whether fresh or several days old, does not decolorise Schardinger's formaldehyde-methylene blue solution. Fresh goat's milk causes only a very slight decomposition of a 1 per cent. hydrogen peroxide solution, and therefore contains only a minute quantity of catalase. Fresh goat's milk decolorises the Rothenfusz, Storch, and benzidine reagents in the same manner as cow's milk.

No distinction with regard to the formaldehyde-methylene blue reaction could be discovered between milks obtained at different stages of milking from sucking and non-sucking goats. The cream of goat's milk does not decolorise the formaldehyde-methylene blue reagent. E. W.

Detection of Adulterants in Saffron by Means of Phosphomolybdic Acid. A. Verda. (*Chem. Zeit.*, 1914, **38**, 325-327.)—The reagent is prepared by dissolving 25 grms. of sodium phosphomolybdate in 90 c.c. of water and adding 20 c.c. of concentrated nitric acid; after eight days the solution is filtered, and is then ready for use; a freshly-prepared solution does not yield satisfactory results. Pure saffron is coloured uniformly green when treated with this reagent, whilst safflower (bastard saffron), Brazil wood, and sandal wood (sanders wood), are unaltered in colour, and marigold-powder, turmeric, and capsicum, are coloured yellowish-green or light green. The presence of about 5 per cent. of these substances may be detected in saffron when the test is applied microchemically; the reaction is due to the crocin present in the saffron. Another reagent, prepared by adding 60 c.c. of concentrated sulphuric acid to 40 c.c. of a 10 per cent. sodium phosphomolybdate solution, yields the following colorations: Saffron, blue; marigold, dirty green; sandal wood, Brazil wood, and logwood, violet; turmeric and capsicum, violet-blue. W. P. S.

Detection of Magnesium Sulphate in Saffron. A. Nestler. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1914, **27**, 388-391.)—If 1 drop of a mixture consisting of equal volumes of saturated magnesium sulphate solution and glycerol is mixed on a microscope slide with 1 drop of chloral hydrate solution, and then covered with a cover-glass, crystals of magnesium sulphate form almost immediately; when the cover-glass is omitted, bubbles of gas are first given off by the mixture, and the crystals appear after a few minutes. Without the addition of chloral hydrate, the magnesium sulphate and glycerol mixture does not yield crystals even when a drop of the mixture is exposed to the atmosphere for many hours. This reaction may be applied to the detection of magnesium sulphate in saffron. A small portion of the sample is mixed with water, filtered, and the filtrate is allowed to evaporate at the ordinary temperature to a syrup; a drop of the latter is then mixed on a microscope slide with a drop of chloral hydrate solution. If crystals form, the mixture on the slide may be washed with alcohol to remove chloral hydrate, colouring matter, etc., and the crystals then further identified as magnesium sulphate by the application of suitable microchemical tests. Of 98 samples of saffron examined by the author during last year, 21 were adulterated with magnesium sulphate (see also ANALYST, 1901, **26**, 272). W. P. S.

Estimation of Salicylic Acid in Jams. H. Serger. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1914, **27**, 319-322.)—The method described by Heintz and Limprich (ANALYST, 1913, **38**, 420) yields trustworthy results when relatively large quantities (50 mgrms. or more) of salicylic acid are being dealt with, but very small amounts of the acid do not yield a coloration with the ferric chloride under the conditions of the test unless 30 c.c. of the petroleum spirit solution of the salicylic acid be employed.

The author prefers the following method as being simpler and more accurate than that of Heintz and Limprich: 20 grms. of the jam containing salicylic acid are heated nearly to boiling with 30 c.c. of water, and filtered; after cooling, 25 grms. of the filtrate are transferred to a stoppered cylinder, 5 c.c. of dilute sulphuric acid are added, and the solution is shaken with 100 c.c. of a mixture of petroleum spirit and chloroform (300 : 200 c.c.). When separation has taken place, 50 c.c. of the petroleum spirit-chloroform layer are transferred to a 100 c.c. cylinder, 1 c.c. of dilute ferric chloride solution is added, and then water up to 100 c.c. The mixture is shaken for one minute, allowed to separate (5 c.c. of ether may be added to accelerate the separation), the petroleum spirit-chloroform layer is drawn off, and the aqueous portion is diluted with water to 100 c.c. The violet coloration of the solution is then compared with a standard prepared by diluting 1 c.c. of ferric chloride solution with 90 c.c. of water, and adding a 0·1 per cent. salicylic acid solution, drop by drop, until the coloration produced is equal to that of the test solution. With quantities of 0·01 to 0·15 grm. of salicylic acid per 100 grms. of jam, the method yields from 90 to 100 per cent. of the amount of acid present. W. P. S.

Determination of the Viscosity of Refined Syrups and Molasses. C. W. R. Powell. (*J. Soc. Chem. Ind.*, 1914, **33**, 238-240.)—A modification of Ostwald's viscosity tube was employed, the larger limb containing about 12 c.c. of the syrup to be examined, and the bulb in the other limb 1 to 1·5 c.c. The capillary in this limb was 6 to 7 mm. long, and had an internal diameter of 2 mm. Consecutive observations of the time of flow for golden syrup, treacle, and molasses, for the same filling of the tube and for repeated fresh fillings, agree very closely, the error varying between about 1 part in 500 to 1 in 4,500. It was found that the temperature coefficients (for 10° C.) for times of flow remain fairly constant for any particular class of liquid, thus enabling the determination to be made at the most convenient temperature, and the viscosity to be subsequently calculated for any desired temperature by means of the predetermined coefficients. Glycerol was adopted as a suitable standard with which to compare the viscosities of the syrups used; there is, therefore, no need to employ an instrument of standard dimensions.

H. F. E. H.

Tobacco Resins. J. von Degrazia. (*Fachl. Mitteil. österr. Tabakregie*, 1913, 109; through *Chem. Zentralbl.*, 1914, I., 1196.)—From the resins obtained from Turkish tobacco, three acids, an alcohol, and, as chief product, a resene, were isolated. O. E. M.

Determination of Vanillin. W. M. Doherty. (*J. and Proc. Roy. Soc. of N. S. Wales*, 1913, **47**, 157.)—When vanillin is treated in a dilute solution with bromine-water and ferrous or ferrous ammonium sulphate, a green colour is produced proportional in its intensity to the quantity of vanillin present, lead hydroxide first being used to decolourise the essence (American official method). The author has been unable to obtain uniform results by this method, and prefers the following, which dispenses with the use of the lead compound: One c.c. of the sample, or its alcoholic distillate, is treated in a small separating funnel with 10 c.c. followed by 5 c.c. of ether. After evaporating off the ether, the aqueous solution is filtered through a moistened filter-paper into a 50 c.c. Nessler glass, and then made up to volume. Ten drops of a freshly prepared saturated solution of bromine-water are then added, followed by 10 drops of a 10 per cent. solution of ferrous sulphate. The colour produced is compared with a 0·2 per cent. solution of pure vanillin (standardised by titration with $\frac{N}{10}$ alcoholic potash: 1 c.c. = ·0152 grm. vanillin), either directly or preferably with a Duboscq colorimeter. The method may be further simplified by adding the bromine and iron solution to the diluted essence, 1 c.c. of essence being diluted to 50 c.c. with water. H. F. E. H.

Difference between the Proportion of Acid and the Degree of Acidity of Wine. C. von der Heide and W. I. Baragiola. (*Zeitsch. angew. Chem.*, 1914, **53**, 249-260.)—Complete chemical and physical examinations were made of two Giesenheimer Fuchsberg wines of the same origin, but different years—viz., 1909 and 1910. The curious fact was established that, although the former contained less titratable acid than the latter, it had a more strongly acid flavour. The chemical analysis showed that the 1909 wine was relatively poor in solid constituents, and was probably derived from decomposing grapes, showing a high proportion of volatile acid. The trouble had been met by frequent and strong sulphuring, with the result that the proportion of sulphuric acid was very high and the alkalinity of the ash low. The 1910 wine had also been strongly sulphured, but less of the sulphurous acid had been converted into sulphuric acid. The estimation of the concentration of hydrogen ions by the sucrose inversion method showed that the relation of the degree of acidity of the 1909 to the 1910 wine was expressed by the ratio 7 : 6, while the ratio of the proportions of acid was 4 : 5. An examination of the physico-chemical balance of the state of combination of the organic acids showed that, whereas the 1910 wine contained considerably more total tartaric acid than the 1909, the proportion of free tartaric acid was very little higher. The free tartaric acid in the 1910 wine must therefore be in a less dissociated condition than that in the 1909, because the proportion of fully and half combined tartaric acid is distinctly higher, and the tartrates depress the dissociation of the free tartaric acid. The sulphuric acid produced by the excessive sulphuring of the 1909 material has had the result of liberating a large proportion of the organic acids from their saline combinations; and although no free sulphuric acid or bisulphates are present, the relative poverty in organic salts has created a high degree of dissociation of the organic acids of the wine. J. F. B.

Estimating Extract in Wine. V. Njegovan. (*Zeitsch. anal. Chem.*, 1914, **53**, 160-165.)—The author agrees with Von der Heide and Schwenk (Analyst, 1912, **37**, 311) that, even for dry wines, the direct method should be abandoned, by "direct method" being understood the German method of evaporation and drying in a water-oven. He does not approve of the proposal of Von der Heide and Schwenk (*loc. cit.*) to estimate the extract of all wines indirectly—that is to say, to calculate the extract from the sp. gr. of the de-alcoholised wine. In his view the French method is the only one hitherto described which gives satisfactory results, but it requires nearly a week for completion, a preliminary drying for four days in a vacuum desiccator being followed by drying to constant weight over phosphoric anhydride, an operation requiring twenty-four to forty-eight hours. The following method gives results in close agreement with those obtained by the French method: About 5 grms. anhydrous sodium sulphate are spread evenly over the bottom of a broad, shallow, stoppered weighing bottle (60 mm. in diameter by 30 mm. high). The bottle and its contents are tared, and 5 c.c. of wine allowed to flow on to the sodium sulphate. The open bottle is placed in a desiccator containing 15 c.c. of concentrated sulphuric acid, and when the contents of the bottle become completely solid (one to two hours), the desiccator is exhausted until the pressure does not exceed 15 mm. After twelve to fourteen hours (overnight), dry air is allowed to enter the desiccator, and the bottle is stoppered and weighed. From the increase in weight of the contents of the weighing bottle, a small correction must be made for traces of water obstinately retained by the sodium sulphate. This correction is determined by a blank experiment with distilled water.

The desiccator must not be exhausted until the mass has set solid, otherwise there may be mechanical loss. With a vacuum of 1 mm. or less, the drying period can be reduced to six hours. G. C. J.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Differentiation between Hen and Duck Egg Albumins for Commercial Purposes. N. Waterman. (*Chem. Weekblad.*, 1914, **11**, 120-126; through *Chem. Zentralbl.*, 1914, I., 1019-1020.)—Considerable quantities of duck egg albumin are imported, mainly from China, in the dry condition, and may be used as an adulteration of hen egg albumin. The following biological method for the differentiation of the two varieties is based on the precipitin reaction: The immunised sera were prepared from rabbits, some immunised against duck albumin, and others against hen albumin. The injections were made three times intraperitoneally and once subcutaneously, in quantities of 6 to 8 c.c., at intervals of seven to ten days. It is important that the albumin contain no yolk. A week after the last injection 20 to 30 c.c. of blood were taken from the ear, yielding about half the quantity of serum. A standard solution of each of the two varieties of albumin was made at a concentration of 1 per 1,000 in physiological salt solution. From these stock solutions a series of highly diluted standards was prepared. The limits of the reactivity of one or the other serum to one or the other albumin were determined by placing 0·1 c.c. of the antiserum in a tube, and adding carefully 1 c.c. of the diluted albumin solutions, so that the two liquids did not mix. After five minutes, if the immunised serum were active, a white ring appeared at the meeting-point of the liquids. The highest dilution at which this ring was produced was taken as the "titre-strength" of the serum. In making the test, five tubes are charged with 0·1 c.c. of hen antiserum, and five with duck antiserum. Standard albumin solutions are made with the two kinds of albumin and with the sample to be tested, each in two concentrations, equal to and twice as strong as the "titre-concentration," and the reaction is observed in the manner described. In an ideal case each of the sera will give no reaction with the other variety of albumin. J. F. B.

Estimation of Chlorine in Blood Serum. S. Gutmann and F. Schlesinger.

(*Biochem. Zeitsch.*, 1914, **60**, 283-286.)—The method of St. v. Bogdandy (Analyst, 1913, **38**, 211) gives results which are too low, owing to the formation of chlorine, which reacts with the silver nitrate, forming soluble silver chlorate. The authors recommend the following modification of the well-known incineration method: The serum (10 c.c.) is treated with 0·5 grm. sodium carbonate, evaporated to dryness on the water-bath, and carefully ignited. The process can be hastened by allowing the partly carbonised residue to cool, adding a few drops of water, and then continuing the ignition. This causes a more rapid disintegration of the carbonaceous residue, and thus facilitates the ignition. It is not necessary to wait until the residue is white; the carbonaceous residue can be extracted with hot water, and the chlorine estimated in the filtrate as silver chloride. E. W.

Adsorption Phenomena in Abderhalden's Dialysis Process. F. Plaut.

(*Münch. med. Wochensch.*, 1914, **61**, 238-241; through *Chem. Zentralbl.*, 1914, I., 920.)—The author has established that in the presence of inorganic, undecomposable substances, such as china clay, barium sulphate, talc, and kieselguhr, an increase in the quantity of hydrolytic products which react with ninhydrin (triketohydrindene hydrate) may be induced. The possibility that small fragments of organs may exert a similar physical influence to that of the inorganic substances cannot be disputed. In those cases where the positive ninhydrin test is to be ascribed to adsorption phenomena, it may be only a matter of non-specific reactions. Hence, from the practical point of view, it follows that a source of error has to be reckoned with, which, if not taken into account, may lead to false clinical deductions. J. F. B.

Utility of the Dialysis Method in Clinical and Biological Problems. E. Abderhalden.

(*Münch. med. Wochensch.*, 1914, **61**, 233-238; through *Chem. Zentralbl.*, 1914, I., 920.)—In the application of protective ferments to substrata in clinical and biological researches, the unreliability of dialysing membranes is a source of error. No material is available which is capable of absolutely excluding proteins and at the same time uniformly permeable to peptones and other protein derivatives. It is possible to work without dialysis by introducing the serum into a test-tube together with the organ, and then removing the albumin of the serum with the organ by coagulation or precipitating agents; the proteolytic products are then examined in the filtrate. This procedure gives good results if every trace of albumin can be successfully separated. Latterly the author has been employing the method of ultra-filtration for the separation of the colloids from the other matters. Another source of error lies in the properties of the substratum employed; those should be chosen which give the most specific reactions. The specific effects of protective ferments will not be decided by using a large number of different organs. The anomaly in the function of an organ is to be sought for in the fact that the cells concerned allow active ferments to pass into the blood-channels. At the same time the substratum also may pass into the blood-system, or the substratum may pass first and be followed by the ferment. The dialysis method in principle has no error; this lies in the carrying out of the experiment. The setting up of numerous control experiments with a large number of organs gives no assurance of the correct result of the main experiments, and is still less a reliable guide on the question of the specific nature of the action of protective ferments. The author describes a dialysing apparatus employed in his work. J. F. B.

Reductase (Fermentation-Reductase) Test. R. Dons. (*Zentralbl. Bakt. u. Parasitenk. I. Abt.*, 1914, **40**, 132; through *Chem. Zentralbl.*, 1914, I., 1223.)—An accurate estimation of the organisms in milk is not possible by the reductase method. The author's investigations show that large quantities of bacteria are necessary to effect the reduction of methylene blue unless they grow freely at 38° C.; even those with an optimum growth at this temperature differ largely in their reducing capacity, which, moreover, varies according to whether raw or sterilised milk is used. Probably the lactic acid forming micrococci and streptococci determine the course of the reaction. This view is supported by the examination of ordinary milk before and after the application of the reductase test. The bacteria of the *coli ærogenes* group effect reduction as rapidly as the lactic acid bacteria in raw, but not in sterilised, milk, but the test gives no indication of their numbers. The test of ordinary contaminated milk is not affected by horse or human excreta in considerable quantities, if the milk is allowed to stand for two hours at ordinary temperatures after their addition. Milk which has been maintained at 100° C. for some time, or at 135° C. for a shorter period, reduces methylene blue; raw milk behaves as such to the test after it has been heated for ten hours at 68°-70° C. Bacterial growth is retarded by methylene blue even in very small quantities. O. E. M.

Detection of Picric Acid in Urine. E. Isnard. (*Ann. Chim. anal.*, 1914, **19**, 100-101.)—Urine containing picric acid exhibits a reddish-yellow colour and resembles that of persons suffering from jaundice. The picric acid may be identified by heating just to boiling 5 c.c. of the urine with an equal volume of saturated sodium hydroxide solution, and then allowing 1 c.c. of ammonium sulphide to flow on to the surface of the mixture. A red-coloured zone is obtained at the junction of the liquids. The test may be rendered more sensitive by extracting the urine with ether, evaporating the ethereal solution, dissolving the residue obtained in a small quantity of water, and testing this solution as described. W. P. S.

Simple Colorimetric Method for the Estimation of Sugar in Urine. E. Riegler. (*Zeitsch. anal. Chem.*, 1914, **53**, 245-249.)—One c.c. of the suitably diluted urine is heated with 10 c.c. of an alkaline solution of glycerol and copper sulphate, and the decrease in the blue coloration of the copper solution is estimated by comparison in the Autenrieth-Königsberger colorimeter. The copper solution, which is perfectly stable, is prepared by mixing a solution of 10 grms. of crystallised copper sulphate in 50 c.c. of water with 10 c.c. of pure glycerol, and diluting the mixture to 1 litre by the addition of a 3 per cent. solution of sodium hydroxide. In use, 10 c.c. of this solution are mixed in a large test-tube with 1 c.c. of the sugar solution and heated over the flame to ebullition; the hot liquid is transferred immediately to a centrifugal tube, centrifuged for one minute, rapidly cooled and compared with the original solution in the colorimeter. The scale divisions of the latter are calibrated by means of a standard solution of pure dextrose previously dried at 110° C. For approximate estimations, 5 c.c. of the alkaline glycerol-copper sulphate solution may be heated to boiling and the sugar solution added to it from a drop-counter, boiling after each drop, until the liquid is decolorised. If albumin be also present it must be removed by precipitation with 5 per cent. β-naphthalene-sulphonic acid solution. J. F. B.

Electrolytic Method for Estimation of Mercury in Urine. H. Palme. (*Zeitsch. Physiol. Chem.*, 1914, **89**, 345-349.)—The organic matter is destroyed by treating the urine (1 litre) with concentrated sulphuric acid (50-75 grms.), heating almost to boiling, and adding powdered potassium permanganate until the colour does not disappear immediately. The hydrated manganese dioxide which separates is dissolved by adding oxalic acid or hydrogen peroxide; in the latter case the excess is decomposed by heating. A solution of 0·1 to 0·2 grm. copper sulphate is then added and the cold liquid treated with hydrogen sulphide for at least forty minutes. The sulphide precipitate is dissolved in dilute sulphuric acid and bromine water, filtered, and the excess of bromine removed. The liquid is then transferred to a platinum basin, preferably with a matt surface, and electrolysed, using a current density of about 0·1 ampère per sq. cm. The copper is deposited as a coherent layer with which any mercury that is present is amalgamated. The basin is then washed with water, alcohol, and ether, dried over potassium hydroxide, and weighed. A perforated lid is then attached to the basin through which a rapid current of dry carbon dioxide is passed, and the contents of the basin heated for some minutes to dull redness. After cooling in a current of carbon dioxide the basin is again weighed. The loss in weight gives the quantity of mercury present. The electrolysis may also be carried out in a glass vessel, using a cathode of platinum foil or gauze, which can then be heated in a glass tube in a current of carbon dioxide, and the volatilised mercury condensed and collected in a capillary tube and identified microscopically or by conversion into the iodide. E. W.

ORGANIC ANALYSIS.

Colorimetric Method for the Estimation of Albumin. E. Riegler. (*Zeitsch. anal. Chem.*, 1914, **53**, 242-245.)—The method is based on the fact that an alkaline solution of albumin dissolves cupric hydroxide, and the intensity of the violet coloration is proportional to the quantity of albumin. A standard solution is prepared in the following manner: 0·5 grm. of pure, dry egg-albumin is placed in a 100-c.c. measuring-flask; about 80 c.c. of a 3 per cent. solution of sodium hydroxide are added, and the mixture is allowed to stand until the albumin is dissolved; 2 c.c. of a 20 per cent. solution of copper sulphate are added, and the mixture is made up to the mark with 3 per cent. sodium hydroxide solution. After shaking, standing for half an hour, and shaking again, the solution is filtered and ready for use. For the estimation of albumin in urine, 10 c.c. of the sample are treated in a graduated centrifugal tube with 5 c.c. of a 5 per cent. solution of β-naphthalenesulphonic acid, which precipitates the albumin. The precipitate is centrifuged, the liquid decanted off, a 3 per cent. solution of sodium hydroxide poured in up to the mark 9·8 c.c., and 4 drops of 20 per cent. copper sulphate solution are added. The mixture is thoroughly shaken, then centrifuged, and 2 c.c. of the violet solution are compared in an Autenrieth-Königsberger colorimeter with the standard solution. If the shades of the two colours are not strictly comparable, a piece of green glass may be placed over the eyepiece, and the two solutions compared as to their intensity of illumination. The method is generally applicable for the estimation of peptone, globulin, nuclein, fibrin, casein, etc., by preparing standard solutions of the respective substances. J. F. B.

Estimation of Alkyl in Combination with Nitrogen. H. Meyer and K. Steiner. (*Monatsh.*, 1914, **35**, 159-161.)—The method of Herzig and Meyer for the estimation of alkyl groups combined with nitrogen depends on the decomposition of the quaternary salt formed with hydriodic acid, and the estimation of the methyl iodide produced. Certain substances, however, split off methylamine, forming the quaternary methyl ammonium iodide, which is not readily decomposed, and low results are obtained. Substances of this type are the methylimides of benzene-polycarboxylic acids, also benzoylmethylamide. In such cases the above method is unsatisfactory, and it is better to saponify the substance with potassium hydroxide, and to estimate the methylamine by absorption in standard acid.

J. F. B.

Comparative Examinations of Raw Caoutchouc and Caoutchouc by Bromination Methods. W. Vaubel and E. Weinerth. (*Gummi. Zeit.*, 1913, **28**, 92-93; through *Chem. Zentralbl.*, 1914, I., 919.)—The authors deal with criticisms raised by Kirchhof and by Schmitz against the Vaubel titration method (*Gummi. Zeit.*, 1912, **26**, 1879). In experiments with unaltered pure Para caoutchouc the bromination method of Budde and Axelrod (*cf.* ANALYST, 1910, **35**, 267; 1911, **36**, 159) and the titration method of Vaubel gave approximately concordant results. In the examination of soft rubber prepared from pure Para caoutchouc the Budde-Axelrod method was the only rapid method which could be employed. Vaubel's titration method requires some further development before it can be regarded as generally applicable with advantage. Various other raw caoutchouc materials showed the presence of resins which readily absorb bromine. J. F. B.

Estimation of Carbon Monoxide by Means of Yellow Mercuric Oxide. L. Moser and O. Schmid. (*Zeitsch. anal. Chem.*, 1914, **53**, 217-233.)—Carbon monoxide is oxidised quantitatively to carbon dioxide by passage over yellow mercuric oxide heated to 100° C.; red mercuric oxide has a far less powerful action. The mercuric oxide is prepared by precipitating a cold saturated solution of mercuric chloride with sodium hydroxide solution. Precipitation should be effected at a low temperature (8° C.), and the precipitate should be protected from the action of light at all stages; it is washed by decantation, collected on a porous clay plate, and dried over sulphuric acid for a week, in the dark. Before it is completely dry it is granulated by rubbing through a fine sieve and then returned to the drying chamber. The apparatus employed for the estimation consists of a U-tube of brown glass, charged with mercuric oxide and heated to 100° C. in a water-bath. The U-tube is connected on the one side to a set of three absorption vessels charged with measured quantities of $\frac{N}{4}$ barium hydroxide solution, and on the other side by means of a capillary T-piece, to a gas burette containing the carbon monoxide over mercury, and to a supply of air purified from carbon dioxide. The rate of passage of gas through the system must not exceed 100 c.c. in twenty minutes. The stream of air is regulated either by pressure or suction, and the introduction of the carbon monoxide through the vertical branch of the T-piece is regulated by dropping mercury from a fine orifice into the compensating tube of the gas burette. After all the carbon monoxide has been passed over, the passage of the current of purified air is continued for forty-five to sixty minutes, whilst the mercuric oxide tube is still maintained at 100° C. The liquid in the last absorption vessel should remain perfectly clear. In the others the excess of barium hydroxide is titrated with standard hydrochloric or oxalic acid in presence of phenolphthaleïn and in a current of purified air. Carbon monoxide may thus be estimated when mixed with hydrogen or methane, the latter gas being unaffected by mercuric oxide at 100° C. Carbon dioxide must be removed by an absorption vessel in front of the mercuric oxide tube. J. F. B.

Detection and Estimation of Gaseous Hydrocarbons in the Water of Mineral Springs. E. Hauser. (*Comptes rend.*, 1914, **158**, 634.)—A known volume of the water is treated with alkali to fix the carbon dioxide and hydrogen sulphide, and shaken with from one-fifth to three-fifths of its volume of air. The presence of combustible gas in the air is shown, and its volume estimated, in the explosion burette. The same water is again shaken up with a similar volume of air, and the combustible gas estimated. If w and w' are the two readings of the explosion burette, the volume of combustible gas in the water taken is $\frac{w^2}{w-w'}$. One extraction with air suffices if the quantity of gas is very small. Results of the analysis of several waters are given. The method gives values for the solubility of methane intermediate between those of Bunsen and of Winckler. O. E. M.

Modifications of the Kjeldahl Method of Estimating Nitrogen. R. Hottinger. (*Biochem. Zeitsch.*, 1914, **60**, 345-352.)—The still-head employed to prevent alkali being carried over is placed in the distillation flask; atmospheric cooling and consequent reflux condensation of ammonia is thus reduced to a minimum. Before distillation the liquid is mixed with one-half its volume of alcohol; it is claimed that this facilitates and hastens the distillation of the ammonia, and that by the time the temperature has risen to within 1° of the boiling-point of water, all the ammonia has been driven off. The paper also contains descriptions and illustrations of other minor modifications. E. W.

Diazonitro Reaction of Vegetable Oils. P. Sisley and Frehse. (*Bull. Soc. Chim.*, 1914, **15**, 295-298.)—The chloride of diazoparanitrobenzene gives pronounced colorations with many vegetable oils. The reagent is prepared by heating 1·4 grms. of *p*-nitraniline with 2·8 c.c. of hydrochloric acid (sp. gr. 1·18) and 10 c.c. of water, diluting the solution with 30 c.c. of water, cooling it in a current of water, adding 8 c.c. of a solution of sodium nitrite, and finally diluting the clear solution to 100 c.c. In applying the test 10 c.c. of the oil are shaken with 5 c.c. of 20 per cent. sodium acetate solution, and a few drops of the reagent. Brown-red to bright red colorations are given by the following oils: arachis, sesame (crude and purified), cottonseed, maize, castor (first and second expression), colza (refined), linseed, nut, poppy, and tung oils. The colour reaches its maximum in about fifteen minutes. When the reaction is negative, the oil assumes an orange-yellow colour owing to the formation of a little phenylnitrosamine and decomposition products of the salt which dissolve in the oil. This is the case with sunflower oil, and with olive oil and olive pulp oil (French, Italian, Spanish, Tunisian), including "huiles tournantes," neutralised oils, and oils containing from 8 to 24 per cent. of acidity. The test is capable of detecting 10 per cent. of arachis oil in olive oil. The reacting constituent appears to be of a phenolic character; it is eliminated by saponification but not by acid treatment. The sensitiveness of the test is affected by rancidity in the case of certain oils.

C. A. M.

Apparatus for the Estimation of Active Oxygen in Soaps and Soap Powders containing Per-Salts. (*Chem. Zeit.*, 1914, **38**, 391.)—In the method proposed by Fuhrmann (ANALYST, 1910, **35**, 372; 1913, **38**, 441) in which the soap is shaken in a stoppered flask with warm water, dilute sulphuric acid, and chloroform, and then titrated with permanganate solution, any portion of the liquid which collects between the stopper and the neck of the flask is liable to be lost when the stopper is removed. In order to avoid this loss, a flask is described having a hollow stopper through which passes a narrow tube. A bulb is blown on the portion of the tube within the flask and the outer end of the tube is closed with the finger when the contents of the flask are shaken. When the finger is removed, the pressure in the flask is released without loss of liquid. W. P. S.

Estimation of Rosin in Varnishes, Oils, and Soaps. H. Wolff and E. Scholze. (*Chem. Zeit.*, 1914, **38**, 369-370; 382-383.)—Comparative estimations of rosin by the various methods suggested for esterification of the fatty acids have shown that the loss of rosin acids is approximately the same in each case, the greatest differences not exceeding 2·5 per cent. The worst results were obtained by Twitchell's original method, whilst those given by Fahrion's method (ANALYST, 1911, **36**, 557), and by the method of esterification of the acids by boiling for ten minutes with alcoholic sulphuric acid were but little better. The most satisfactory of the methods were based on esterification of the fatty acids by boiling for two minutes with alcoholic sulphuric acid, as follows: From 2 to 5 grms. of the mixture of fatty and rosin acids are dissolved in 10 to 20 c.c. of absolute methyl alcohol, and the solution treated with 5 to 10 c.c. of a solution of 1 part of sulphuric acid in 4 parts of methyl alcohol, and boiled for two minutes under a reflux condenser. The liquid is then mixed with five to ten times its volume of 7 to 10 per cent. sodium chloride solution, and the fatty acid esters and rosin acids extracted with ether, or a mixture of ether and petroleum spirit. The united extracts are washed with dilute sodium chloride solution, alcohol added, and the mixture titrated with $\frac{N}{2}$ alcoholic potassium hydroxide solution. A mean acid value of 160 is taken for the rosin acids, and a correction of 1·5 per cent. allowed for fatty acids that have escaped esterification. The percentage of rosin acids is then obtained by the formula—

$$x = \frac{a \cdot 17{\cdot}76}{m} - 1{\cdot}5,$$

where a represents the number of c.c. of potassium hydroxide solution, and m the weight of the mixture taken. The result multiplied by 1·07 gives approximately the amount of rosin. *Gravimetric Method.*—From 2 to 5 grms. are esterified as described above, and, after neutralisation, the ethereal solution is treated with an excess of 1 to 2 c.c. of the alcoholic alkali, and repeatedly washed. The washings and soap solution are concentrated to a small volume, acidified and shaken out two or three times with equal volumes of concentrated sodium chloride solution. The ethereal layer is dried with fused sodium sulphate, the ether distilled, the cold residue dissolved in 10 c.c. of absolute ethyl alcohol, and the solution treated with 5 to 10 c.c. of a mixture of 1 part of sulphuric acid and 4 parts of ethyl alcohol, and allowed to stand for one and a half to two hours at the ordinary temperature. An addition of seven to ten times the volume of concentrated sodium chloride solution is now made to the mixture, which is then extracted two or three times with ether. The united extracts are washed with dilute sodium chloride solution, dried with anhydrous sodium sulphate, and distilled. The percentage residue of rosin acids is multiplied by 1·07 to obtain the amount of rosin. The tabulated results show that this method gives results at least as accurate as any previous method, while it is simpler and much more rapid.
C. A. M.

Estimation of Free Alkali in Commercial Soda and Soaps by the Barium Chloride Method. A. Kling, V. Genin, and D. Florentin. (*Bull. Soc. Chim.*, 1914, **15**, 200-205.)—Experiments here described show that the estimation of free alkali in soda and soap by precipitating carbonate, stearate, and oleate, as barium salts and titrating the filtrate with standard acid, is inaccurate in the presence of alkali silicates or borates, since barium silicate and barium borate are appreciably soluble and affect turmeric and phenolphthaleïn in the same way as caustic alkali. On the other hand, accurate results are obtained by precipitating in a 50 per cent. alcoholic solution, in which all the barium salts, including the borate and silicate, are insoluble, while sodium and barium hydroxides are readily dissolved. Fifty c.c. of a 1 per cent. solution of the soap or soda are treated with a slight excess of 10 per cent. barium chloride solution, the liquid diluted with its own volume of 95 per cent. alcohol, allowed to stand for some time, and filtered, and an aliquot portion of the filtrate titrated at boiling temperature with $\frac{N}{20}$ sulphuric acid (turmeric as indicator). A sample of commercial sodium carbonate containing 25 per cent. of sodium silicate showed by this method 0·004 grm. of caustic alkali, whereas estimation in an aqueous medium gave 0·032 grm. per litre of the solution. C. A. M.

INORGANIC ANALYSIS.

Estimation of Traces of Arsenic. L. Moreau and E. Vinet. (*Comptes rend.*, 1914, **158**, 869-871.)—The method, which is applicable to the estimation of such quantities of arsenic as 0·001 mgrm., depends on the generation of arsine and the decomposition of this by means of silver nitrate solution, with the production of a mirror of silver which can be compared with other mirrors prepared in a similar manner from known quantities of arsenic.

As the method is intended to estimate quantities as small as 0·0005 mgrm., a quantity which might be present in 10 grms. of the purest zinc obtainable, the authors use only 0·5 grm. zinc for generating the arsine, and sweep the latter out of the apparatus by means of a current of purified hydrogen (*cf.* McGowan and Floris, *Report of Royal Commission on Arsenical Poisoning*, 1903, *Appendix* 22). The apparatus consists of a hydrogen generator, a gas-washing apparatus in which the main current of hydrogen is freed from any arsine by contact with a dilute solution of silver nitrate, the arsine-generating apparatus, and the tube for the deposition of the silver mirror. The arsine-generating apparatus consists of a U-tube 1 cm. in diameter and 15 cm. high. Through the bung closing one limb there pass the stem of a tap-funnel and a tube for introducing the stream of purified hydrogen. The other limb is closed by a one-hole bung, which carries a double right-angled bend, the outer end of which is lightly plugged with absorbent cotton and connected by a short piece of rubber-tubing to the mirror tube, the adjacent end of which is similarly plugged. The mirror tube is of 5 mm. diameter, and bent twice at right angles, the horizontal portion being 3 to 4 cm. long, and the outer vertical limb about 15 cm. long. The arsine-generating apparatus being charged with 0·5 grm. of freshly platinised zinc and a few drops of water, and the apparatus connected up, air is expelled by setting the hydrogen generator in operation, and 0·5 c.c. of a faintly acid $\frac{N}{10}$ solution of silver nitrate is run into the open end of the mirror tube. A long thread of glass is also pushed into this tube, and serves to reduce the mechanical loss of liquid particles by the gas current. About 0·5 c.c. of dilute (1 : 5) sulphuric acid is run into the arsine apparatus, the gas current is regulated to about fifteen to twenty bubbles per minute, and the apparatus allowed to operate for ten minutes, during which time no mirror should appear. The liquid to be examined (which should be small in bulk with the particular apparatus described) is next introduced into the arsine apparatus by means of the tap-funnel, and if arsenic is present in sensible amount, a mirror will begin to make its appearance within five minutes. The solution of silver nitrate oscillates with the passage of the bubbles of gas, and the mirror makes its appearance at the point on the tube to which the liquid comes back after the passage of each bubble. The rubber connections permit one to incline the mirror tube and vary the position of this point, and this affords a convenient means of determining whether all the arsenic has been expelled from the apparatus. In the contrary event, a second mirror appears. Mirror tubes before use should be washed with *aqua regia*, water, and alcohol successively and dried. At the end of an experiment they should be disconnected in such a way that the mirror is not wetted by the silver nitrate solution, allowed to drain, exposed to the light, and compared with standards preferably next day, as the mirrors become blacker and more lustrous with time.

It is said that the mirrors cannot be confounded with deposits of Ag_3Sb, which have a more pronounced colour and less metallic lustre. They are still more readily distinguished from deposits of silver sulphide, but sulphides can readily be eliminated in the preparation of the sample, and this should always be done. G. C. J.

Differentiation of Natural from Artificial Cryolite. (*Sprechsaal*, 1914, **47**, 133; through *Chem. Zentralbl.*, 1914, I., 1218.)—Suspensions of natural cryolite in distilled water are unaffected by boiling, and settle on standing, leaving a clear liquid which does not react with baryta, silver nitrate, and phenolphthaleïn, and leaves no residue on evaporation. The artificial substance is decomposed on boiling with water, and yields a turbid emulsion on suspension, while its solution gives an intense violet-red coloration with phenolphthaleïn, and a white residue on evaporation. Lead acetate produces a precipitate with both, soluble in excess of the reagent only in the case of the natural product. Hydrochloric acid dissolves aluminium from both, but much more from the artificial than from the natural substance. The differences in behaviour on cooling in test-tubes, stated by Coen and Biazzo (*Soc. Chim. Ital.*, February, 1911) to be characteristic, were found to be uncertain, but the micro-chemical data were confirmed. O. E. M.

Occurrence of Fluorine in Reagents. P. Carles. (*Ann. Chim. anal.*, 1914, **19**, 101-104.)—The author finds that, whilst sulphuric, hydrochloric, and acetic acids are usually free from fluorine, nitric acid always contains this substance. Fluorine is also usually present in potassium nitrate, sodium nitrate, barium hydroxide, barium acetate, and ammonium carbonate, and may occur in sodium bicarbonate and potassium bicarbonate. W. P. S.

Errors in Gas Analysis due to assuming that the Molecular Volumes of all Gases are alike. G. A. Burrell and F. M. Seibert. (*U. S. Bureau of Mines, Technical Paper*, **54**, pp. 16.)—In determining the purity of certain combustible gases by the method of slow combustion, the authors found that in some analyses the volume of carbon dioxide and the contraction produced by the combustion did not agree with the theoretical value. Also, in analysing some natural gases by the same method, the paraffin hydrocarbons present totalled over 100 per cent. These discrepancies have been traced to the fact that carbon dioxide does not behave as a perfect gas. Wohl endeavoured to establish the error which might enter into the results of a gas analysis by the assumption that molecular volumes of all gases were exactly equal, and he derived certain expressions for correcting the results of such analysis (*Ber.*, 1904, **37**, 429). The authors, however, agree with Haber (*Thermodynamics of Technical Gas Reactions,* 1906, 306) that Wohl's expressions are only applicable in special cases, his correction for carbon dioxide, for example, being applicable only when carbon dioxide constitutes 95 per cent. or more of the gas measured. When the partial pressure of the carbon dioxide is less than 30 per cent. of the total pressure, the mixture behaves so nearly like a perfect gas that no correction is necessary, and Haber consequently recommended that the partial pressure of the carbon dioxide should always be kept low by dilution, as is done in explosion analyses for other reasons. The authors point out, however, that this may imply small readings and considerable magnification of errors. In such cases—analyses of natural gas—they prefer to submit nearly the whole of the sample to slow combustion with the smallest possible excess of oxygen, and to make suitable corrections. A number of tables of corrections, calculated from well authenticated density determinations, are given. The most important of them are derived from the following numbers, which represent the ratio of the theoretical to the observed density at 760 mm. and 0° C. of each of the gases named: methane 0·999, ethane 0·990, ethylene 0·983, acetylene 0·992, carbon dioxide 0·994. The ratios are substantially the same at 20° C. as at 0° C., but of course approach unity in proportion as the partial pressure of the gas is reduced from 760 mm. G. C. J.

Separation and Estimation of Beryllium. A. Kling and E. Gelin. (*Bull. Soc. Chim.*, 1914, **15**, 205-210.)—The basic acetate of beryllium [$BeO_4(CH_3CO_2)_3$] is readily soluble in chloroform, and can be distilled unchanged at the ordinary pressure (b.-pt., 330° C.). It is not possible to effect a quantitative separation of beryllium from iron and aluminium by treatment with chloroform, since a little of the acetate of beryllium remains entangled with the other acetates, whilst, on the other hand, the solvent dissolves some iron. Quantitative results may be obtained by volatilising the basic beryllium acetate from the basic acetates of iron and aluminium. The solution of the mixed chlorides is treated with a slight excess of ammonia, the precipitated hydroxides dissolved in warm dilute nitric acid, the solution filtered, and evaporated to dryness in a platinum boat, and the residue ignited over an alcohol flame until a porous mass is obtained. This is cooled and moistened thoroughly with glacial acetic acid, the excess of which is subsequently evaporated at 105° to 110° C., this process being repeated several times. The boat containing the basic acetates is placed in a glass tube (45 to 50 cms. long, and 4 to 5 cms. in diameter), one end of which is closed with a rubber stopper, through which passes a tube connected with a small mercury manometer. The manometer communicates with a tube containing glacial acetic acid, while the large tube is connected with a water pump. A reduced atmosphere always charged with acetic acid vapours is maintained within the tube, which is gently heated to about 160° to 170° C., until a deposit of beryllium acetate begins to appear. This temperature is maintained for about four hours, after which it is gradually increased to 250° C., and kept at that point for another hour, the vacuum being meanwhile maintained. After cooling, air is admitted, the basic beryllium acetate dissolved off the walls of the tube and dissolved in fuming nitric acid, the solution evaporated to dryness, and the residue dried at

110° C. until constant in weight. The residue in the boat is again treated with glacial acetic acid and the sublimation repeated to separate the last traces of beryllium. Finally, the residual iron and aluminium acetates are dissolved in nitric acid, the solution evaporated, and the residue ignited until constant in weight. The results are very accurate in the case of mixtures containing at least 50 per cent. of beryllium. When a smaller proportion is present the mixture of the oxides should be treated with a concentrated solution of sodium carbonate, which will dissolve all the beryllium, and only slight amounts of the iron oxide and alumina. The solution is evaporated and the beryllium separated from the residue as described. C. A. M.

Volumetric Estimation of Mercuric Chloride. W. Stüwe. (*Chem. Zeit.*, 1914, **38**, 320.)—The method depends on reduction to metal by means of hydrazine sulphate and subsequent estimation of the excess of reducing agent employed. The principal reaction is effected in presence of alkali bicarbonate, and the excess of hydrazine sulphate is determined iodimetrically in presence of acetic acid and sodium acetate—that is to say, in absence of free mineral acid, which is set free when hydrazine sulphate reacts with iodine.

An approximately 1 per cent. solution of hydrazine sulphate is standardised by adding to a measured quantity, contained in a stoppered flask, some sodium acetate and a measured and more than sufficient quantity of $\frac{N}{10}$ iodine solution. (A 1 per cent. solution of hydrazine sulphate is about 0·3 *N*.) After fifteen minutes, the excess of iodine is titrated with $\frac{N}{10}$ thiosulphate.

To conduct an analysis, the solution of mercuric chloride, which may conveniently contain about 0·2 grm. $HgCl_2$, is introduced into a 100 c.c. stoppered flask, mixed with a measured and more than sufficient quantity (10 c.c.) of the hydrazine sulphate solution, a little sodium bicarbonate is added, and the flask is set in a bath of lukewarm water, as the reaction requires an appreciable time, which is less at slightly elevated temperatures. The reaction is usually complete in about fifteen minutes, and the subsidence of the precipitated mercury is an indication that this point has been reached. The contents of the flask are then cooled, made up to 100 c.c., filtered through a hardened filter (S. and S., No. 602), and 50 c.c. of the filtrate are transferred to a stoppered flask, acidified with acetic acid, mixed with some sodium acetate and a measured volume of $\frac{N}{10}$ iodine solution, more than sufficient to decompose the hydrazine sulphate remaining. The flask is stoppered, and after fifteen minutes the excess of iodine is titrated with $\frac{N}{10}$ thiosulphate. The relation $N_2H_4 = 4I = 2HgCl_2$ sufficiently explains all the reactions involved, but for analytical purposes only the relation $2I = HgCl_2$ needs to be considered. G. C. J.

Reaction of Nitrates with Formic Acid. U. Molinari. (*Staz. sperim. agrar. Ital.*, 1914, **47**, 24; through *Chem. Zentralbl.*, 1914, I., 1221.)—In testing the method of Quartaroli (Analyst, 1911, **36**, 468) the most suitable concentration for the formic acid was found to be 95 per cent., and the reaction could conveniently be carried out in a Schiff nitrometer. In no case were theoretical values obtained; with potassium nitrate the results varied from 91·33 to 94·55, and with barium nitrate from 89·97 to 92·72, per cent. of the theoretical. Analysis of the products showed that the reaction does not take the course indicated by Quartaroli—

$$2KNO_3 + 6HCOOH = N_2O + 4CO_2 + 5H_2O + 2HCOOK,$$

but that nitric oxide is also formed, and that the yield of carbon dioxide, nitrous oxide, and nitric oxide varies. Hence the method cannot yield accurate results.

O. E. M.

Field Method for Determining Dissolved Oxygen in Water. J. Miller.

(*J. Soc. Chem. Ind.*, 1914, **33**, 185-186.)—The method is that of Linossier (*J. Soc. Chem. Ind.*, 1891, **10**, 726), adapted to field conditions. To 50 c.c. of the water contained in 100 c.c. Nessler jar are added 5 c.c. of alkaline tartrate, such as is used for Fehling's solution, and 1 drop of phenosafranine (1 in 2,000 of water). From a 10 c.c. graduated pipette, a solution of ferrous sulphate (0·22 grm. of $FeSO_4$ and 1 c.c. of concentrated sulphuric acid in 100 c.c.) is run in just below the surface of the liquid, stirring gently with the pipette, which should have a very small outlet hole, until the colour is discharged, looking through the cylinder horizontally. The slight coloration formed at the surface of the liquid does not interfere if the stirring is done gently. The pipette reading gives the c.c. of oxygen per litre. Theoretically, the iron solution should have contained 0·25 grm. to do this, but in practice the value given above (0·22 grm.) gives correct results. Each experimenter should standardise the ferrous sulphate solution, employing water of known oxygen content, such as distilled water shaken with air till saturated at a known temperature. The method gives results about 5 per cent. higher than Winkler's. The ferrous sulphate keeps well, and experiment showed that waters containing up to 60 parts of sodium nitrite per 100,000 had no disturbing effect on the method, which cannot, however, be used in presence of cyanides.

H. F. E. H.

Volumetric Estimation of Phosphates in the Ash of Food Materials. B. Pfyl.

(*Arbeit. Kaiserl. Gesundheitsamte*, 1914, **47**, 1-44.)—Methods which have been proposed previously for the titration of phosphates (*cf.* ANALYST, 1908, **33**, 273) are not free from objection, since, even with pure phosphates, the results obtained are not absolutely accurate. In the case of very small quantities of phosphate, the error is correspondingly small, and useful results may be obtained, but with large quantities of phosphate the error becomes appreciable. The author has therefore investigated the conditions which influence the titration of phosphates, and shows that trustworthy results may be obtained by the following procedure in which the phosphate solution is rendered neutral towards methyl-orange, treated with calcium chloride, and then titrated until just alkaline to phenolphthaleïn. The reaction according to the equation—

$$2NaH_2PO_4 + 3CaCl_2 \longrightarrow Ca_3(PO_4)_2 + 4HCl + 2NaCl,$$

is only complete when the solubility of tricalcium phosphate and the formation of monocalcium phosphate ($CaHPO_4$) and basic calcium phosphate [$Ca_3(PO_4)_2.Ca(OH)_2$] are prevented by the addition of a sufficient quantity of calcium chloride; the temperature and time of reaction are also of importance.

Titration of Pure Phosphates.—The phosphate solution, measuring about 25 c.c., and containing not more than 50 mgrms. of P_2O_5, is rendered slightly acid towards methyl-orange, and then titrated with $\frac{N}{10}$ alkali solution until the coloration changes to yellow; 30 c.c. of a 40 per cent. neutral calcium chloride solution are then added, the mixture is heated to boiling, cooled to 14° C., 2 drops of a 1 per cent. phenolphthaleïn solution are added, and $\frac{N}{10}$ alkali solution is added slowly until the solution is coloured distinctly red. The vessel containing the mixture is now closed, immersed for two hours in water at a temperature of 14° C., and the titration then continued until the red coloration reappears (as a rule, the red coloration disappears during the cooling). From the total quantity of $\frac{N}{10}$ alkali solution used there must be deducted 1 per cent. of the volume (a correction for the quantity of carbonate present), and the remainder is multiplied by 3·55 to give mgrms. of P_2O_5. Quantities of P_2O_5 up to 89 mgrms. may be titrated in a similar way if the mixture is cooled to 4° C. before the phenolphthaleïn is added, and then further cooled in ice-water for sixteen hours before the final titration.

Titration of Phosphates in Ashes.—The substance is ignited gently after the addition of alkali, and the ash is treated with a small quantity of water and a few drops of concentrated hydrochloric acid; 1 drop of 30 per cent. hydrogen peroxide is added, and, when the ferric oxide has dissolved, the mixture is treated with $\frac{N}{4}$ alkali solution until nearly neutral to methyl-orange. The mixture is now heated on the water-bath for fifteen minutes, cooled, one drop of $\frac{N}{1}$ alkali solution is added, and the precipitated ferric and aluminium phosphates are collected on a filter, and washed twice with 4 c.c. of water; the filter and precipitate are then warmed for twenty minutes (not more) on a water-bath with 30 c.c. of a 40 per cent. neutral sodium citrate solution. The filtrate is meanwhile rendered neutral to methyl-orange, then added to the citrate solution of the ferric and aluminium phosphates, 1 drop of phenolphthaleïn solution is added, and the mixture is titrated with $\frac{N}{1}$ alkali solution, the titration being completed after the neutralised solution has been cooled in ice-water for twenty minutes. The ferric and aluminium phosphates may also be titrated separately after their solution has been cooled to 0° C. as just described. If the ash contains borates, the quantity of boric acid present may be estimated by a further titration of the solution used in the estimation of the phosphates; mannitol is added to the solution, and the titration continued until the solution is just alkaline to phenolphthaleïn. W. P. S.

Gravimetric Estimation of Selenium. J. Meyer. (*Zeitsch. anal. Chem.*, 1914, **53**, 145-154.)—It is found that solutions of selenious acid cannot be evaporated without loss, and that in presence of hydrochloric acid the loss may be very large indeed, and is not measurably reduced by the presence simultaneously of an alkali chloride. On the other hand, it is found that it is not necessary to expel nitric acid from solutions of selenious acid before proceeding to precipitate the selenium in the elementary condition; it suffices to neutralise the nitric acid with ammonia, and re-acidify with hydrochloric acid. The most convenient reagent for reducing selenious acid to selenium is hydrazine hydrate, but special means must be taken to guard against mechanical loss owing to the rapid evolution of nitrogen, and to the fact that the selenium separates at first in a very fine state of division, and only goes over into the grey crystalline form after a period of heating. A special apparatus, designed by Jannek in the course of his work on the atomic weight of selenium (*Zeitsch. anorg. Chem.*, 1913, **83**, 51) is figured in the paper. It consists of a flask connected by a ground joint to a spray trap, which is connected by another ground joint to a bubbler containing a little water. The flask, which has a thistle funnel sealed into it, serves also for effecting the solution of selenides in nitric acid, another operation which is apt to be accompanied by mechanical loss.

Substances in which it is desired to estimate selenium are dissolved in nitric acid, and the solution is freed, if necessary, from elements which might interfere with the estimation, and then neutralised with ammonia. It is then acidified with hydrochloric acid, the presence of which greatly increases the speed with which red amorphous selenium passes into the grey crystalline modification, and introduced into the apparatus described. It is heated to boiling, and a sufficient quantity of 10 per cent. hydrazine hydrate solution is added very slowly. The use of stronger solutions of hydrazine may give rise to trouble. When the separated selenium has a grey crystalline appearance, and further additions of hydrazine develop no red colour in the solution, heating is discontinued. With the apparatus described, the removal of the flame causes the water in the bubbler to flow back into the flask, rinsing the spray trap on its way. The selenium is finally filtered off on a Gooch crucible, dried in a toluene bath, and weighed. G. C J.

Determination of Melting-Points of Silicates. H. Leitmeier. (*Tschermaks min. u. petr. Mitt.*, 1914 [II.], **31**, 536-544 ; through *Chem. Zentralbl.*, 1914, I., 1109-1110.)—The observed melting-points of silicates depend on other factors besides the chemical composition of the mineral. Unless the temperature be raised very slowly, no melting-point, but only a melting interval, can be recorded, and this interval is the greater when the particles are larger. In the author's view more importance attaches to records of the temperature at which the crystalline mineral loses its definitely crystalline structure than to records of alleged melting-point, as the former figure is less dependent on experimental conditions.

By greatly prolonging the period of heating and reducing the rate of rise of temperature, very much lower melting-points than any hitherto published have been obtained, and some of these are almost sharp, whilst in others the interval is a much closer one than is usually recorded. For example, a sample of adular began to melt at 1,145±5° C., and after forty-eight hours at this temperature was completely liquid. Other workers, using more ordinary methods, have recorded the interval 1,240° to 1,350° for this material. Similarly augite, for which the melting interval 1,210° to 1,250° C. has been recorded, has been found to melt between 1,145° and and 1,160° when the temperature was raised from 1,145° at about 1° per hour. Other examples are given in the paper. G. C. J.

Estimation of Zinc in Coinage Bronze by Volatilisation. T. K. Rose. (*J. Soc. Chem. Ind.*, 1914, **33**, 170-172.)—The bronze is heated to about 1,375° C. in gas carbon crucibles covered with charcoal in a salamander cover pot for two hours, and the residue weighed. As many as eight or sixteen carbon crucibles may be treated in one charge, while to insure uniform heating the cover pot is rotated in the gas injector furnace through 90° every fifteen minutes. This high temperature is necessary, as the loss is insignificant at the temperature of a gas assay muffle furnace (1,200° C.). The use of check assay pieces containing a known percentage of zinc is necessary, as the loss varies considerably with the temperature. Coinage bronze contains 1 per cent. of zinc, the usual loss found by volatilisation being 1·2 per cent., while the average difference between two duplicate assays is 0·02 to 0·03 per cent. By means of the check assay on a trial plate of known zinc percentage, the necessary correction can be applied to the sample under investigation. There appears to be no reason why the method should not be applicable to brasses and bronzes containing any proportion of zinc; but in cases containing high proportions, it is advisable to reduce the zinc to about 5 per cent. by boiling off at 1,000° C. so as to avoid loss of copper by volatilisation. The following losses of copper and tin occurred with different proportions of zinc.

Alloy.			Loss by Volatilisation, per Cent.	Loss of Copper and Tin by Volatilisation.	
Copper, per Cent.	Tin, per Cent.	Zinc, per Cent.		On whole Alloy, per Cent.	On Copper and Tin, per Cent.
96·0	4	0·0	0·14	0·14	0·14
95·0	4	1·0	1·22	0·22	0·22
92·35	0	7·26	7·69	0·43	0·43
8·75	0	91·25	91·55	0·30	3·44

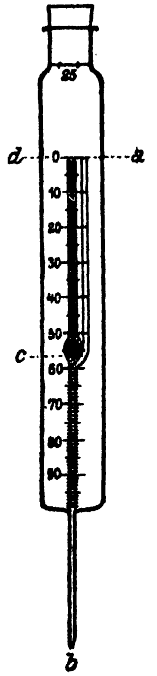
Duplicate determinations are in about as close agreement with high percentages of zinc as with low, and by using pieces of the 1 per cent. trial plate as check assays, results from brasses are obtained which are only about 0·1 to 0·2 per cent. too high in zinc. The presence of cadmium or zinc oxide will of course cause the value for zinc to appear too high, while the error due to the retention of zinc amounts to about 0·03 per cent. as a rule, the original percentage of zinc making no difference in this respect; the use of a check assay will remove this error and also that caused by loss of copper and tin by volatilisation (usually about 0·2 per cent.). A convenient weight to take for the determination is 1 grm. of the alloy. H. F. E. H.

APPARATUS, ETC.

Efficient Boiling-Rod. G. Torossian. (*J. Amer. Chem. Soc.*, 1914, **36**, 513-514.)—Pieces of an alundum crucible or porous porcelain plate are crushed in a mortar to a coarse powder, the pieces being about 0·5 mm. in diameter. The fragments are heated to redness in a crucible, and a piece of glass rod 10 to 12 c.c. long, heated until it softens, is dipped into the alundum particles, which will adhere to the rod. This is repeated two or three times until sufficient particles adhere to the rod. Another form of rod is prepared by fusing on to the end of a glass rod a small rectangular or triangular piece of alundum, the rod being then bent in such a manner that the fused piece will lie on the bottom of the beaker or flask. The boiling-rod should be placed in the solution when it reaches the boiling-point and not before.

H. F. E. H.

Apparatus for the Approximate Estimation of Free Carbon Dioxide in Mineral Waters. R. Fresenius and L. Grünhut. (*Zeitsch. anal. Chem.*, 1914, **53**, 265-274.)—A simple apparatus for the approximate estimation of dissolved gases in mineral waters is shown in the figure. This consists of a shaking-tube, 24 cm. long and 30 mm. wide, closed at the bottom, and reduced at the top to a neck, which is closed by a soft rubber stopper. At the bottom of the neck is a mark indicating exactly 125 c.c., and the surface of the rubber stopper must be level with this mark. Inside the wide tube a narrow, open tube, *a-b*, is fused through the lower end. This tube is bent at *c* as shown, and a thermometer is fused on at the bend as a continuation of the axis. The outer tube is graduated with the zero at *a*, situated so that at the level of the opening of the small tube the contents of the larger tube are exactly 100 c.c. The instrument is filled with a sample of the mineral water up to this point; the sample should be taken by immersing the instrument in the water without agitating the latter. The level of the water is allowed to adjust itself through the overflow tube by holding the instrument in a vertical position. The cork is inserted as far as the mark, and the instrument is turned to an inclined position, with the neck downwards. The cork is held in with one hand, and the orifice *b* is closed by a finger of the other. The instrument is shaken vigorously twenty times, so that the pressure of the liberated gas accumulating in the instrument forces a jet of water out of *b* when the finger is removed. This is repeated until no more water is ejected; the instrument is then returned to the vertical position, and the volume of water displaced is read off. The results for carbon dioxide at $t°$ C. and 760 mm. are calculated by the formula:

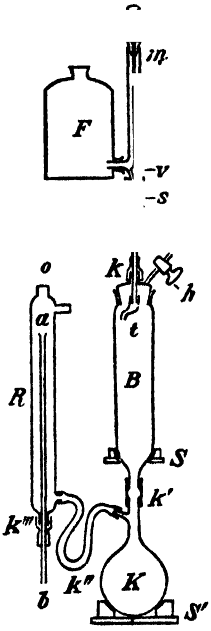


$$CO_2 \text{ (c.c. per litre)} = 10\,C + \frac{C}{V+C} \cdot \frac{1100\,a\,(273+t)}{273},$$

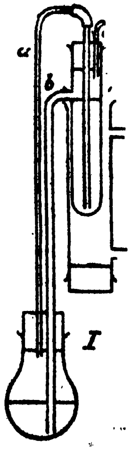
in which V is the volume of air originally contained in the filled instrument (generally 25 c.c.), C is the volume of carbon dioxide measured by the water displaced, and a is the absorption coefficient per 1 c.c. of water at 0° C. and 760 mm. The value $\frac{1100\,a\,(273+t)}{t}$ may be calculated for various temperatures in the form of a table. The results when compared with accurate gravimetric estimations show an error within ±5 per cent.

J. F. B.

Gas Generator for Permanent Use. U. Kreusler. (*Zeitsch. anal. Chem.*, 1914, **53**, 234-241.)—The apparatus designed for the generation of gases in the laboratory—*e.g.*, sulphuretted hydrogen, carbon dioxide, hydrogen, etc.—is illustrated in the figure. The charge of acid is contained in the bottle *F*, of about 6 litres capacity, which is connected with the regulating outflow tube *A* by means of a luted joint packed with a mixture of gutta-percha and paraffin wax. The bottom end of the tube *A* is connected by means of the ground-glass joint with a tube *c*, which is so narrow that the acid completely fills it owing to capillarity. The flow of the acid is regulated by the conical glass valve *v*, which is operated from above by means of a glass rod. This rod has a spiral thread of glass fused around its upper portion, where it passes through the split cork *m*, so that the valve may be controlled by a screwing action. The solid substance—*e.g.*, iron sulphide, is placed in the cylindrical glass vessel *B*, which contains about 4 kilos of it. At the bottom of *B* is placed a spirally-wound funnel of stout copper or silver wire, which prevents solid matter from falling through. The cork support *S* is permanently fixed to the wall, but has a slit through which the apparatus can be removed. The flask *K* serves to collect the mud which settles out in the course of use, and the clear liquid flows through the rubber tube *k*, into the overflow and pressure regulator *R*, consisting of a Liebig's condenser with central overflow pipe adjustable in height according to the pressure desired. The small adapter *t* on the end of the acid tube is for preventing the drops of acid always falling in one spot; by rotating the tube *c*, the flow of acid may be distributed in whichever direction it may seem desirable. When in use, the liquid rises in the pressure regulator and drops out as fast as the acid comes in; thus a constant pressure is maintained. The gas is taken off from the cock *h*. J. F. B.



Apparatus for Ether Extraction of Liquids. J. J. L. Zwikker. (*Pharmaceutisch Weekblad*, 1914, **51**, 21-23; through *Chem. Zentralbl.*, 1914, I., 1046.)—The illustration is almost self-explanatory. The flask holds the ether, the vapour of which rises through the tube, *a*, and is conducted to the bottom of the liquid to be extracted contained in the test-tube. Fig. II. represents on a larger scale the lower end of the tube, which conveys the ether vapour to the bottom of the liquid; and Fig. III. is a still further enlarged plan view of this arrangement for securing uniform distribution of the bubbles of ether vapour. The ether condenses as it passes up through the liquid, and automatically flows back to the flask through *b*. G. C. J.



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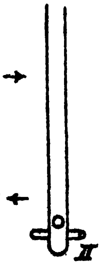
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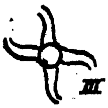
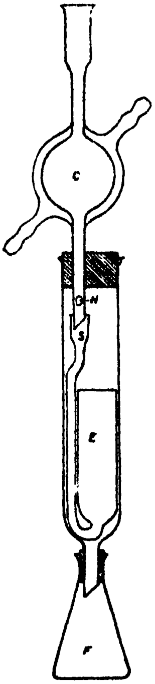

III

New Extraction Apparatus. S. N. Pinkus. (*Biochem. Zeitsch.*, 1914, **60**, 311-313.)—The apparatus, an illustration of which is given in the original, is a modification of the Soxhlet, designed for the extraction of substances at low temperatures, and especially of proteins, when it is desired to avoid coagulation.

E. W.



Extraction Apparatus specially designed for Solvents of High Specific Gravity. C. Budde. (*J. Soc. Chem. Ind.*, 1914, **33**, 184-185.)—When chloroform or carbon tetrachloride are used to extract wax or fat, the solution so obtained has a lower sp. gr. than the original solvent, and thus tends to accumulate in the upper part of the extraction apparatus. This is obviously the reverse of what should happen for efficient extraction, and the solvent in such cases should be supplied from the bottom, and not, as in the ordinary Soxhlet apparatus, from the top. In the apparatus figured (made by Messrs. Brady and Martin, Newcastle-on-Tyne) the vapours of the solvent pass outside the extractor, *E* (kept at the boiling temperature), through the hole *H* in the condenser tube, into the condenser, whence the condensed solvent flows through the side tube *S* into the bottom of the extractor. The solution of wax or fat overflows from the top of the extractor down into the flask *F*. The apparatus may with advantage be used for the extraction of liquids such as aqueous solutions or emulsions, with solvents of lower sp. gr., such as ether. In such cases the side tube *S* must be high enough to allow the ether to force its way through the water into the extractor.

H. F. E. H.

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REVIEWS.

A Textbook of Quantitative Chemical Analysis. By A. C. Cumming, D.Sc., and S. A. Kay, D.Sc. London: Gurney and Jackson. Edinburgh: Oliver and Boyd, 1913. Pp. xi + 382. Price 7s. 6d.

This book is primarily intended for University and college students, and the subject-matter is so arranged that "some knowledge of the principles of quantitative analysis may be acquired by a practical study of the subjects included in Parts I., II. and III." (General Principles, Volumetric Analysis, Gravimetric Analysis), "and that the further requirements of those who are making a special study of chemistry should be met by the later portion of the book" (Colorimetric Methods, Systematic Quantitative Analysis, Simple Ores and Alloys, Gas Analysis, Water Analysis, Quantitative Analysis of Organic Substances and Determination of Molecular Weights.

Volumetric analysis is placed before gravimetric, the authors in their preface expressing the view that "a student should receive a thorough training in volumetric analysis, even if the time remaining at his disposal permits of little or no gravimetric work." Although the importance of an early training in volumetric methods is universally recognised, the above proposition strikes one as being somewhat sweeping. The relegation of gravimetric work to a secondary position, with the intimation that if the student is pressed for time it may be omitted altogether, is surely calculated to give him a false impression of the fundamental principles of analytical work.

As far as the actual subject-matter of the book is concerned, this is treated in a clear and concise manner, and the instructions as to manipulative details, together with, for the most part, adequate explanations of the principles involved, are obviously the outcome of considerable experience in teaching. The average student will, however, be puzzled by the reference on p. 45 to "sulphuric acid and . . . sodium hydroxide, both of which ordinarily contain varying amounts of carbonate."

The arrangement of the section on systematic quantitative analysis is open to criticism. The "system" adopted is an alphabetical one and is intended to include all the common elements and radicles, together with the methods for their separation and determination. The information given is in many cases exceedingly scanty, especially where methods of separation are concerned. Under several headings—*e.g.*, ammonium, calcium, chloride and sulphate—the only information consists of cross references to other parts of the book. If these paragraphs were inserted in this section for the sake of completeness, it is difficult to understand the reason for the omission of iodide, cyanide, nitrate and nitrite, although methods for their estimation are to be found in other parts of the book. Moreover, the reader will look in vain for any mention of borate or fluoride, both of which might have been deemed worthy of inclusion in a section intended to "prove a useful index to quantitative methods in general." Such alphabetical arrangement of bases and acids as is needed is fully supplied by the index at the end of the book, and a system more in accordance with the chemical relationships of the substances dealt with would have avoided an appearance of confusion in the section as a whole.

Several features which do not ordinarily appear in textbooks of this kind will be welcomed, such as the electrolytic determination of cadmium and lead, and of copper and nickel with a rotating cathode; the colorimetric estimation of the relative acidity or alkalinity of natural water by comparison against standard phosphate solutions, with azolitmin as indicator; and the estimation of halogen in organic compounds by decomposition with sodium in presence of alcohol.

In the section on water analysis it is to be regretted that the well-known Griess-Ilosvay method for the estimation of nitrites is omitted in favour of the one based on the liberation of iodine from potassium iodide.

The observations on p. 350 as to the difficulty of obtaining temperature measurements accurate to $\frac{1}{100}$th degree with the Beckmann thermometer are very much to the point, in view of the frequency with which one sees such measurements recorded to the third place of decimals.

The illustrations of apparatus have been specially drawn for the book and are in most cases very well done. An appendix is added giving various useful tables, details as to the preparation of reagents and typical analyses of a few minerals, and the book is adequately indexed. G. W. MONIER-WILLIAMS.

THE FÆCES OF CHILDREN AND ADULTS. By P. J. CAMMIDGE, M.D. London: J. Wright and Sons, 1914. Price 17s. 6d. net.

The author's original intention was to translate and edit Dr. Adolf F. Hecht's book, "Die Fæces des Säuglings und des Kindes," but on consideration he decided to publish a book of wider scope, embracing his own experimental work of many years' duration, but making use of Dr. Hecht's book and also that of Schmidt and Strasburger on "Fæces des Menschen."

The main feature of the volume is the extreme thoroughness which the author has devoted to a subject which is unfortunately too often neglected.

In the two chapters, the one on the chemical examination of the fresh, the other on that of the dried fæces, the methods of detection and estimation of the various constituents are detailed in an exceedingly practical manner.

The tables of fat analysis of the fæces in various diseases, if read by themselves, would not greatly assist in the diagnosis of alimentary canal and allied affections; but, as the author rightly points out, the percentages of the "total fat," "unsoaped fat," and "combined fatty acids," must be considered along with the clinical manifestations from which the patient is suffering, and in this way are of considerable assistance.

Chapters with excellent illustrations are devoted to the bacteriological examination of the fæces, to the animal parasites, and to the calculi and concretions found in the alimentary canal.

At the end of each chapter is a bibliography of all the recent work relating to the subject-matter.

Various useful dietaries are added in an Appendix. The book is one which can be recommended as eminently practical both to the medical practitioner and to the chemical pathologist, and as being the most complete yet published in the English language.

E. BURSTAL.

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INSTITUTE OF CHEMISTRY.

PASS LIST: MARCH EXAMINATIONS, 1914.

OF ten candidates who presented themselves for the Intermediate Examination, nine passed: A. S. Carlos, B.Sc. (Lond.), C. G. Collins, (Miss) Gwen Dyer, Nat. Sci. Tripos (Cantab.), F. L. Elliott, H. S. Foster, J. J. Geake, G. Harding, J. W. Sewill, B.A. (Cantab.), and G. T. Shipston. Of fourteen candidates who presented themselves for the Final Examination, eight passed. In the Branch of Organic Chemistry: P. K. Dutt, M.A., B.Sc. (Calcutta), J. W. Harris, B.Sc. (Lond.), and E. Mather, B.Sc. (Lond.); in the Branch of the Chemistry (and Microscopy) of Food and Drugs, Fertilisers and Feeding Stuffs, Soils and Water: P. S. Arup, C. W. McHugo, F. S. Thurston, B.Sc. (Lond.), J. A. F. Wilkinson, B.Sc. (Lond.), and F. Wright, B.Sc. (Leeds).

JUNE, 1914. Vol. XXXIX., No. 459.

THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

An ordinary meeting of the Society was held on Wednesday evening, May 6, in the Chemical Society's Rooms, Burlington House. The President, Mr. A. Chaston Chapman, F.I.C., occupied the chair.

The minutes of the previous ordinary meeting were read and confirmed.

Certificates of proposal for election to membership in favour of Messrs. William Roscoe Hardwick, B.Sc. (Vict.), B.Sc. (Liv.), F.I.C., 13, Batavia Buildings, Hackins Hey, Liverpool, analytical and consulting chemist; Harold Fletcher Hills, Commercial Gas Works, Stepney, E., chemist to the Commercial Gas Company; and Robert Hindle Kay, 72, Manor Road, Stoke Newington, N., analytical chemist to the Crown Perfumery Co., Homerton, were read for the first time.

Messrs. L. H. D. Acland, B.A., W. A. Gibbings, F. H. Lees, F.I.C., and W. H. Woodcock were elected members of the Society.

The following papers were read: "The Detection of Castor Oil Seeds," by G. D. Lander, D.Sc., F.I.C., and J. J. Geake; "The Composition of Milk," by H. Droop Richmond, F.I.C.; "The Action of Weak Acids on Soluble Fluorides," by P. A. Ellis Richards, F.I.C.; and "Notes on Sharps," by J. F. Liverseege, F.I.C., and G. D. Elsdon, B.Sc., A.I.C.

A simple form of Fat Extractor was exhibited by Mr. G. A. Stokes.

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THE COMPOSITION OF MILK AND MILK-PRODUCTS.

By H. DROOP RICHMOND, F.I.C.

(*Read at the Meeting, May* 6, 1914.)

The present communication deals with the results obtained during 1913 in the laboratory of the Aylesbury Dairy Company.

The total number of milk samples examined was 38,582, and these are divided into several series; the largest and most important is that referring to the milk as received from the farms, and the mean composition is given in Table I.:

TABLE I.

Average Composition of Milk during 1913.

Month.	Morning Milk.				Evening Milk.				Mean.			
	Sp. Gr.	Total Solids.	Fat.	Solids-not-Fat.	Sp. Gr.	Total Solids.	Fat.	Solids-not-Fat.	Sp. Gr.	Total Solids.	Fat.	Solids-not-Fat.
		%	%	%		%	%	%		%	%	%
January ...	1·0318	12·46	3·64	8·82	1·0316	12·65	3·85	8·80	1·0317	12·56	3·75	8·81
February ...	1·0320	12·39	3·55	8·84	1·0315	12·56	3·76	8·80	1·0318	12·48	3·66	8·82
March ...	1·0320	12·30	3·46	8·84	1·0317	12·46	3·67	8·79	1·0319	12·38	3·57	8·81
April	1·0319	12·18	3·38	8·80	1·0315	12·39	3·64	8·75	1·0317	12·28	3·51	8·77
May	1·0323	12·18	3·31	8·87	1·0318	12·52	3·69	8·83	1·0320	12·35	3·50	8·8
June	1·0324	12·12	3·24	8·88	1·0317	12·43	3·63	8·80	1·0321	12·27	3·43	8·84
July	1·0318	12·07	3·32	8·75	1·0312	12·40	3·71	8·69	1·0315	12·24	3·52	8·72
August ...	1·0315	12·18	3·47	8·71	1·0310	12·50	3·85	8·65	1·0312	12·34	3·66	8·68
September ...	1·0315	12·37	3·62	8·75	1·0311	12·80	4·06	8·74	1·0313	12·59	3·84	8·75
October ...	1·0318	12·49	3·68	8·81	1·0315	12·85	4·02	8·83	1·0316	12·67	3·85	8·82
November ...	1·0319	12·65	3·78	8·87	1·0317	12·93	4·04	8·89	1·0318	12·79	3·91	8·88
December .	1·0319	12·73	3·84	8·89	1·0317	12·90	4·01	8·89	1·0318	12·82	3·93	8·89
Average ...	1·0319	12·34	3·52	8·82	1·0315	12·62	3·83	8·79	1·0317	12·48	3·67	8·81

The morning and evening milks have been kept separate, and the numerical mean of the two is given. This table, representing 19,807 samples, is the most nearly representative of the mean composition of milk during 1913. The difference between the numerical average and the true average, which should take into account the relative bulks of the morning and evening milks, is exceedingly small; but an indication of the amount of difference may be obtained by a comparison with another series, containing 11,325 samples, which represents the milk sent out from the chief and branch depots. The monthly figures differ but very slightly from the figures in Table I., and the average percentage of fat is 3·66, as against 3·67; in the second series the differences due to the varying quantities of morning and evening milks are eliminated. Three smaller series, containing 1,180, 2,348, and 1,573 samples respectively, confirm the general average.

The average percentage of fat found (3·67) is very slightly lower than in 1912, and this is practically entirely due to a falling off during the month of November (3·91 per cent. as against 4·10 per cent.). Last year I remarked that the percentage of fat during November was the highest found for many years. The amount of fat was higher during the first five months in 1912 than in 1913, but during June, July, and August it was lower. The difference between the percentages of fat found in the morning and evening milks (0·31) is rather less than usual.

During 1913 there was the usual fall in solids-not-fat in the months of July, August, and September, but no very low percentages were noticed.

In continuation of the tables given in former years, I give the percentages of morning milks falling below 3·0 per cent.; in 1913 it was only in May, June, and

July that any appreciable proportion of low fats were noticed, and they were practically absent from the evening milks.

TABLE II.

Month.	2·9 to 3·0.	2·8 to 2·9.	2·7 to 2·8.	Below 2·7.
May	3·5	1·9	0·9	0·4
June	4·6	2·2	0·8	0·9
July	2·6	2·0	0·9	0·6

During 1913 the systematic determination of the aldehyde figure, from which the percentage of proteins was calculated, was continued, as also of ash, and the results of these, as well as the percentages of milk-sugar (by difference) appear below:

TABLE III.

Month.	Solids-not-Fat.	Milk-Sugar.	Proteins.	Ash.
January	8·81	4·57	3·52	0·74
February	8·82	4·65	3·42	0·75
March	8·81	4·68	3·38	0·75
April	8·77	4·61	3·43	0·73
May	8·85	4·56	3·53	0·76
June	8·84	4·55	3·53	0·76
July	8·72	4·50	3·47	0·75
August	8·68	4·50	3·43	0·75
September...	8·75	4·58	3·43	0·74
October	8·82	4·52	3·55	0·75
November...	8·88	4·58	3·55	0·75
December	8·89	4·66	3·48	0·75

A sample of goat's milk and one of human milk were examined; the results are—

	Goat's Milk.	Human Milk.
Specific gravity	1·0337	1·0279
Total solids	12·66	8·28
Fat	3·42	0·97
Milk-sugar	4·71	5·80
Proteins	3·66	1·12
Ash	0·77	0·39
Acidity	17·5°	1·3°

The sample of human milk was somewhat abnormal; the fat was very low, and the milk-sugar and proteins were low also, while the amount of ash was higher than that usually found. The aldehyde figure was determined and found to be only 3·3°, giving a factor of 0·34 for conversion into proteins. The sample was nearly transparent, and was evidently abnormal with respect to the nature of the proteins, but was too small for further investigation.

The Aldehyde Figure of Cream.—The percentages of fat and aldehyde figure have been determined in a number of cream samples, with the following results:

Fat. Per Cent.	Aldehyde Figure.	Aldehyde Figure, $\frac{100}{}$
60·2	8·1°	20·4°
47·9	10·2°	19·6°
47·5	11·0°	21·0°
47·5	10·6°	20·2°
46·7	12·3°	23·1°
45·8	11·8°	21·8°
45·0	12·0°	21·8°
44·2	11·8°	21·1°
42·5	12·3°	21·4°
41·8	11·8°	20·3°
39·8	11·9°	19·8°
38·4	13·2°	21·4°
38·0	11·8°	19·0°
24·2	15·7°	20·7°
		Average 20·8°

It is seen that the aldehyde figure of the cream devoid of fat is practically the same as that of milk devoid of fat.

This figure is of use for detecting rapidly whether a cream has been watered or not, for should a cream be low in fat, due to the addition of milk, no alteration of the figure would take place, while watering would lower it.

Two watered creams gave results as under:

Fat. Per Cent.	Aldehyde Figure.	Aldehyde Figure, $\times \frac{100}{100 - \text{Fat}}$.
38·2	10·0°	16·7°
25·4	12·4°	16·6°

DISCUSSION.

Mr. **EMBREY** thought it desirable that figures such as these should be accompanied by a full statement of the sources from which the samples were derived. Otherwise, those cases in which the figures were abnormally low might be quoted as a standard for genuine milk, although he presumed that Mr. Richmond did not in every case possess direct evidence that the milk was exactly as it came from the cows. It was a fact, for which he (Mr. Embrey) could vouch from recent personal experience, that bad handling might result in a considerable loss of fat after a long railway journey. It was very difficult to ascertain the average composition of human milk. During the past few years he had examined a considerable number of samples, and had found the fat to vary from 1 per cent. to 8 per cent., the only constant feature being the lowness of the sugar.

Mr. **REVIS** drew attention to the fact that the average figures obtained by him for the past year were practically identical, month by month, with those obtained by Mr. Richmond. For this reason, he was of the opinion that such average figures were of comparatively little use in individual cases, and, in fact, might lead to considerable misapprehension, as it appeared to him that if only a large enough number of samples were examined, covering a large area of production, practically the same average would always be obtained. It would also be noticed that the average figures for 1912 and 1913 were identical, but the monthly variations of the milk during the two years were distinctly different. These average figures were no criteria by which to judge individual consignments.

Dr. **VOELCKER** said that he believed it had been shown that the idea that churns ought to be ventilated in transit was a mistaken one, and that there was no real reason why churns should not be so sealed that none of their contents could be lost. He thought that Mr. Richmond did good service by publishing his figures annually, inasmuch as they showed that without much difficulty a higher standard could be attained than was now generally accepted. At the same time, milk of poor quality sometimes occurred entirely as the result of purely natural causes.

Mr. **E. M. HAWKINS** asked whether the 1913 samples included any in which the non-fatty solids were below 8·5 per cent.

Dr. **MONIER-WILLIAMS** said that in dealing, some time ago, for statistical purposes, with the results of several thousand analyses of milk supplied by different farmers to certain large dairies, he had found that the curves obtained by plotting out the percentage number of samples containing different proportions of fat were more characteristic of the quality of the milk as a whole, and gave more valuable information, than if averages only were considered. He had observed that very great differences often occurred in the quality of the milk from different farms in the same district.

Mr. **RICHMOND** said that he quite realised the desirability of a full statement of the conditions under which the samples were obtained, but if such a statement were given every year it would involve a good deal of repetition, and, as a matter of fact, in the earlier years, and from time to time later, pretty full particulars had been given by Dr. Vieth and by himself. It was difficult to condense the results of so many analyses into a few figures, and he had been thinking that it might be worth while, in addition to giving the averages, to give what was known mathematically as the "dispersion," and so give some idea of the extent of the variations. It was a great advantage to plot the results in curves, and the "dispersion" could be obtained in that way by noting the points of inflexion.

The samples that were low in non-fatty solids were extremely few—much fewer than was the case in 1911. The question as to whether any selection was made of the samples had been raised on previous occasions, when he had explained that no selection at all was made unless there was distinct evidence of interference with the milk—such, for instance, as deficiency in the quantity contained in the churn on arrival, as compared with the quantity invoiced. There was no doubt as to the wide variations in the constituents of human milk, even to a larger extent than Mr. Embrey had mentioned. It was curious, however, that such variations as occurred in cow's milk at different periods of the milking, did not occur to the same extent in the case of human milk. In conjunction with Professor Carter, of Birmingham, he had investigated this point some years ago, and had found that the proportion of fat before and after suckling was almost identical, differences only occurring when the secretion was very deficient in quantity. His experience was that, as a general rule, human milk was more constant in composition than Mr. Embrey's experience indicated, when lactation had become normal. Samples of normal human milk were, however, not so readily obtained for analysis as samples taken during the first few days after birth. The milk was then more of the nature of colostrum; but after about a fortnight its composition became normal, the milk-sugar being in most cases between 6 and 7 per cent., and the proteins between 1 and 2 per cent., while the fat became quite as constant as in individual cow's milk.

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THE ACTION OF WEAK ACIDS ON SOLUBLE FLUORIDES.

By P. A. ELLIS RICHARDS, F.I.C.

(*Read at the Meeting, May* 6, 1914.)

The fluorides of the alkali metals have recently come into prominence as suggested preservatives for articles of food.

In Reports to the Local Government Board by Drs. MacFadden and Monier-Williams (Food Reports No. 17 and Report on the Work of Inspectors of Foods for the year 1912-13) reference is made to one or two preparations of this nature advertised under proprietary names, with the statement that the preservative is of a perfectly harmless character.

As, so far, these preservatives have been found mainly in milk, cream, and butter, the action, if any, of lactic acid upon soluble fluorides becomes a matter of some importance. With a view to obtaining information on this point, as a preliminary experiment, some pure concentrated lactic acid was allowed to act upon a little powdered sodium fluoride contained in a lead capsule covered with a waxed glass marked in the usual way. Evolution of hydrofluoric acid speedily occurred, and distinct etching of the glass was obtained, the latter being naturally increased when the vessel was warmed.

Dilute solutions of lactic acid of 5, 2 and 1 per cent. strength respectively were all found to act in a similiar manner upon the fluorides of sodium and potassium, liberating hydrofluoric acid. In the latter, and subsequent experiments, watch-glasses waxed and marked on the concave surfaces were employed to hold the mixed solutions.

The positive result obtained with lactic acid suggested that probably other organic acids might act in a similar manner.

The acids most likely to be found in foods—acetic, butyric, tartaric, malic, and citric acids—were experimented with in turn, and in each case, employing a concentration of 1 per cent. of acid and 1 per cent. of sodium fluoride, distinct etching resulted.

I may perhaps mention that all the acids employed were carefully tested before use to insure their purity as regards traces of mineral acids, whilst both the sodium and potassium fluorides were free from ammonium fluoride. Control experiments of the same duration, employing similar apparatus containing the fluoride solution only, gave no sign of etching.

The ease with which hydrofluoric acid was liberated by the organic acids mentioned suggested the possibility of carbonic acid gas producing the same result. To determine this point, tubes or bottles, waxed and marked internally, were employed to hold the sodium fluoride solutions. The carbon dioxide, after passage through washing-bottles, was allowed to bubble through the liquid from time to time for an hour. Hydrofluoric acid was again liberated, the etching, although definite, being not quite so pronounced as in the previous experiments.

By allowing solution of a fluoride saturated with carbonic acid gas to remain in contact with the glass for some hours a much deeper etching was obtained. Hydrofluoric acid was also liberated by formic, salicylic, and benzoic acids, but not by boric acid. Lactic acid and the other acids mentioned above, that gave positive results with sodium and potassium fluorides, failed to do so with calcium fluoride, even when the latter was employed in a fine state of subdivision.

Dilute solutions of the mineral acids—sulphuric, nitric, hydrochloric, hydrobromic, and hydriodic acids—as one would expect, all liberated hydrofluoric acid from sodium and potassium fluorides.

The two salts differ in some respects, potassium fluoride being deliquescent, whilst sodium fluoride is a dry powder. Both slowly absorb carbon dioxide from the atmosphere, hydrofluoric acid being set free, and this may possibly account for the action of the fluorides of the alkali metals upon the glass of bottles in which they have been long stored.

As a result of these experiments it is evident that both sodium and potassium fluorides are easily decomposed by the majority of weak acids with which they may be brought in contact, and this fact emphasises their undesirability as preservatives for food of any kind.

DISCUSSION.

The PRESIDENT remarked that all the alkaline fluorides were more or less readily hydrolysed in aqueous solution, and presumably any addition of acid would increase the dissociation, so that a little hydrofluoric acid would be formed. This would be driven off on heating, and a further quantity would be produced, and so on. With boric acid a boro-fluoride would probably be formed. On the addition of boric acid to an acid fluoride solution, a strongly alkaline solution was obtained, and the same, of course, happened with silica.

Mr. E. R. BOLTON said that the fact that fluorides were very effective in preventing the growth of mould in butter and margarine was no doubt due to formation of hydrofluoric acid in the manner which the President had mentioned, the fluoride being acted upon by the fatty acid resulting from hydrolysis of the fat by the mould.

Dr. LANDER remarked that the liability of fluorides to hydrolytic dissociation furnished an interesting problem in connection with the periodic law, since theoretically one would anticipate less hydrolysis with fluorides than with chlorides.

Dr. LESSING drew attention to the well-known experiment in which ultramarine was decolorised in a solution of hydrofluoric acid by passing carbonic acid into it.

The PRESIDENT remarked that hydrofluoric acid did not fall into line with the chlorides and bromides, its formula at ordinary temperatures being, not HF, but H_2F_2.

Dr. MONIER-WILLIAMS said that fluorides had, on the strength of certain experiments on mice and other animals, frequently been stated to be harmless when used as food preservatives. It seemed, however, extremely dangerous to draw such a conclusion in view of certain experiments which had been carried out on the Continent on the amount of fluorine ingested and excreted by dogs fed for a prolonged period on food containing small amounts of sodium fluoride—amounts considerably less than would be required to cause gastric irritation. It would appear that a certain quantity of the fluorine thus ingested remained in the body, associated with, and possibly replacing to a certain extent, the phosphates of the bones and teeth, and that in the course of a year or more comparatively large quantities might thus be taken up. Until these results had been confirmed or disproved, it was obvious that caution must be exercised in drawing conclusions from isolated experiments of a few days' duration upon animals, and that the possibility of sodium fluoride acting as a cumulative poison was by no means excluded.

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NOTES ON "SHARPS."

By J. F. LIVERSEEGE, F.I.C., and G. D. ELSDON, B.Sc., A.I.C.

(Read at the Meeting, May 6, 1914.)

In the milling of wheat, about 70 per cent. is obtained as flour, about 10 per cent. as "sharps," and about 20 per cent. as bran.

A sample of sharps which yielded 7·02 per cent. of ash, of which 0·70 per cent. was insoluble in HCl, and which contained 3·19 per cent. of calcium sulphate ($CaSO_4$, $2H_2O$), raised the question as to what were the permissible limits for ash and sulphate in genuine sharps.

The Agricultural Analyst for Derbyshire has recently informed us that the ash in 17 samples he had examined varied between 1·60 and 3·84 per cent., the average being 2·91. In 13 samples examined by the Public Analyst for Warwickshire the maximum ash was 4·75 per cent., and the ash insoluble in HCl 0·37 per cent.

Lawes and Gilbert (*J. Chem. Soc.*, 1884, **45**, 305) published an extensive series of analyses of wheat grown at Rothamstead. Grain grown on ten plots of land during twenty years was analysed. The average amount of sulphate calculated as gypsum was 0·06 per 100 parts of dry grain. The lowest figure was 0·002 per cent., and the highest 0·16 per cent., which was grown on a plot which had been regularly treated with ammonium sulphate. One hundred and ten analyses by Wolff, of winter wheat, gave an average on the dry grain of 0·02 per cent.

We found that a mixture of five samples of commercial flour yielded sulphate equivalent to 0·05 per cent. of gypsum. A single analysis by Bell ("Chemistry of Food," ii. 88) gives 0·02 per cent. on the dry flour. We know of no published results indicating the amount of sulphate in "sharps," and we think the following analyses will be of interest. The sulphate was determined as $BaSO_4$ on the ash of 20 grms. of sharps. Calcium was determined by the method of Fresenius. The ash of 20 grms. of "sharps" was dissolved in dilute hydrochloric acid, and ammonia added, drop by drop, until a slight permanent precipitate was formed. This was redissolved in the smallest quantity of hydrochloric acid, excess of solid ammonium oxalate added, and then a considerable quantity of solid sodium acetate. The calcium was weighed as oxide. In each case the calcium was in excess of the equivalent of the sulphate present.

The amount of sulphate calculated as gypsum on the samples as received, containing 9·8 to 13·7 per cent. of water, varied from 0·02 to 3·19 per cent., and there seems little doubt that some of the samples contain gypsum either purposely added, or possibly in some cases accidentally present. We think that a limit of 0·5 per cent. is ample to allow for any accidental contamination, and that samples containing more than this should be considered unsatisfactory.

The amount of ash insoluble in acid in No. 10 was large, owing to the presence of oat-husk in the sample, and further analyses were made of it and of the others to obtain comparative figures. 1·3 per cent. of this sample failed to pass a No. 12 sieve. Of No. 7, 1 per cent., which was chiefly pearl barley, was left behind. The other samples only left a mere trace on this sieve. In the table is given the total amount of each sample which would not go through a No. 20 sieve, the holes of which are practically 1 sq. mm. in area. Determinations of the percentages of ash insoluble in acid and of albuminoids were made on the fractions. The sample containing oat-husk contained the smallest percentage of albuminoids in the fraction, and the largest amount of ash insoluble in acid. Determination of fibre in the "sharps" gave no useful result, 5·1 being present, as compared with from 4·6 to 6·8 in three other samples which were free from oat-husk.

ANALYSES OF "SHARPS."

Number.	1*	2	3	4	5	6	7	8	9	10*	11*	12	13†	14‡
Moisture	12·1	12·2	10·3	13·6	11·6	13·5	13·7	11·4	11·6	13·0	13·8	9·8	10·2	13·4
Albuminoids	14·3	16·4	14·8	16·1	18·2	15·7	14·1	19·2	16·7	13·6	15·1	15·3	13·9	13·5
Ether extract...	4·3	4·8	3·9	3·5	4·4	4·6	3·1	4·1	4·4	3·3	4·1	3·4	3·9	4·3
Total ash	2·63	3·54	4·97	4·14	3·94	4·06	4·86	5·36	5·46	4·04	4·64	5·04	5·84	7·02
Ash insoluble (HCl)... ...	0·11	0·22	0·97	0·18	0·12	0·46	0·60	0·46	0·34	0·82	0·32	0·56	0·41	0·70
Calcium (Ca)	—	0·12	—	0·14	0·13	0·13	0·36	0·58	0·62	0·46	0·70	0·82	1·15	1·14
Sulphate (SO_4)	0·01	0·03	0·06	0·11	0·12	0·12	0·23	0·23	0·39	0·62	0·79	0·76	1·77	1·78
Gypsum ($CaSO_4$, $2H_2O$) ...	0·02	0·06	0·12	0·21	0·22	0·22	0·41	0·42	0·71	1·12	1·42	1·38	3·17	3·19
Number 20 fraction	0·0	1·8	3·0	1·9	1·6	1·7	6·3	2·6	3·0	4·6	3·5	3·5	3·8	6·8
Per cent. of albuminoids in No. 20 fraction	—	—	—	—	15·0	14·6	13·6	13·8	—	7·8	12·5	—	—	13·6
Per cent. ash insoluble in HCl in No. 20 fraction ...	—	0·22	0·78	0·52	0·31	0·15	0·60	0·76	0·87	2·32	0·27	1·01	0·69	0·45

* Same vendor. † Pearl barley and a little oat-husk present. ‡ Oat-husk present.

NOTE BY EDITOR.—If Nos. 1-6 represent genuine samples of sharps, there would appear to be no reason for allowing so high a figure as 0·5 per cent. of gypsum, and this would be borne out by Lawes and Gilbert's figures and the others quoted above. In the case of Nos. 10-14, and more especially Nos. 13 and 14, some explanation further than that of the presence of a little oat-husk is necessary to account for the high gypsum figure. This applies similarly, though to less extent, to Nos. 7-9, and we should be disposed to consider these as suspicious, and 10–14 as undoubtedly impure.

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Analysis of Adhesive Soluble Substances. G. Armani and J. Barboni. (*Annali Chim. Appl.*, 1914, **1**, 138-142.)—Gum arabic is detected by the peroxydase reaction, and glue by giving precipitates with tannin and with an alcoholic solution of lead acetate. If glue is found, the solution is hydrolysed with hydrochloric acid and tested for reducing sugars, derived from gum, by means of an alkaline solution of orthonitrophenylpropiolic acid. Tests for furfural from dextrins, soluble gums, etc., are applied to the solution after removal of glue by tannin. Soluble gums, of which gum arabic is typical, contain arabin, which is hydrolysed to equal parts of arabinose and galactose. Bassorah gum contains about 11 per cent. of arabin, and an insoluble portion of bassorin. Gum tragacanth contains more arabin than bassorin, whilst the semi-soluble gums, such as rosaceous gums, contains cerasin and arabin, the former being transformed into the latter, and then dissolving in hot water. This property distinguishes cerasin from bassorin. The presence of 5 per cent. of gums may be detected by the cherry-red coloration which is obtained when the yellow solution obtained in the benzidine acetate test is treated with 30 per cent. potassium hydroxide solution until a precipitate appears, and then with acetic acid until the precipitate dissolves. C. A. M.

Aporeine and its Salts. V. Pavesi. (*Gazz. Chim. Ital.*, 1914, **44**, 398-405.) —The poppy, *Papaver dubium*, contains an alkaloid, aporeine, $C_{18}H_{16}NO_2$, which may be purified by recrystallisation from petroleum spirit. The crystals melt at 88° to 89° C., yielding a greenish-yellow fluorescent liquid, which turns brown at 260° C., and becomes a black mass at 280° to 290° C. A characteristic property of the alkaloid, when dissolved in certain solvents, is that it shows a bluish fluorescence very similar to that of quinine salts. Its specific rotation $[\alpha]_D$ in 95 per cent. alcohol was $+75{\cdot}19$ at 15° C. Hydrochloric acid dissolves the alkaloid without materially altering it. The neutral (normal) sulphate melts at 70° to 75° C., and, when exposed to the air and light, decomposes, forming a reddish-brown powder.

C. A. M.

Detection of Beef and Mutton Fats in Lard by Bömer's Method. H. Sprinkmeyer and A. Diedrichs. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1914, **27**, 571-581.)—The application of Bömer's method (ANALYST, 1914, 84) to numerous samples of genuine lard, beef and mutton fats, and mixtures of the same, yielded results showing that the method is capable of detecting the presence of 10 per cent. of beef or mutton fats in lard; in most cases 5 per cent. may be detected. In the case of lards rendered by the authors the difference between the m.-pts. of the least soluble glycerides and those of the fatty acids obtained from the same varied from 4·4° to 7·4° C., whilst the value MG + 2d (m.-pt. of the glyceride + twice the difference of the m.-pts.) ranged from 73·1 to 76·5. With beef and mutton fats the m.-pt. difference varied from 0·8° to 1·2°, and the value MG + 2d from 65·2 to 67·3. In the case of mixtures of lard and beef or mutton fats, and containing from 5 to 10 per cent. of the latter fats, the value MG + 2d was always below 72, and in the greater number of cases below 70 even with the mixtures containing only 5 per cent. of beef or mutton fat. The presence of hydrogenated (hardened) oils or fats in lard affects the value MG + 2d to the same extent as does the presence of beef fat.

W. P. S.

Rapid Estimation of Fat in Cheese, Butter, and Cream. K. Kropat. (*Arch. Pharm.*, 1914, **252**, 76-82.)

Cheese.—A weighed quantity (2 to 5 grms.) of the sample is heated over a flame with 5 c.c. of hydrochloric acid (25 per cent.) until dissolved. The solution is mixed with 3 to 5 c.c. of alcohol, cooled, and shaken with 25 c.c. of ether until separate layers are obtained, when the extraction is completed with 25 c.c. of petroleum spirit. The separating funnel is allowed to stand for about fifteen minutes, after which 1 to 1·5 grms. of gum tragacanth and 3 to 5 c.c. of water are introduced, and the funnel rotated until the gum swells up, and then shaken until a clear ethereal layer is obtained. This is drawn off, the residue of coagulated gum, etc., washed with 15 c.c. of petroleum spirit, the extract and washings evaporated, and the residue dried for two hours and weighed.

Butter.—One grm. of the sample is heated with 3 to 5 c.c. of water, 3 to 5 c.c. of alcohol added, the mixture cooled, and the fat extracted with 20 c.c. of ether and 20 c.c. of petroleum spirit. The casein is then coagulated by the addition of 1 grm. of gum tragacanth and 1 to 3 c.c. of water, and the extract of fat separated and evaporated as described.

Cream.—A mixture of 2 to 5 grms. of the cream with 3 c.c. of ammonia solution and 3 to 5 c.c. of alcohol is shaken in a separating funnel with 20 c.c. of ether and 20 c.c. of petroleum spirit. In the case of sour cream 3 to 4 c.c. of water or ammonia solution are added after the shaking to make the fat dissolve completely. The flask is left for ten to fifteen minutes, and after the addition of 1 to 1·5 grms. of gum tragacanth and 3 to 4 c.c. of water (if not already added), it is rotated and shaken as described. The ethereal layer and washings (15 c.c. of petroleum spirit) are evaporated, and the residue of fat dried and weighed. C. A. M.

Detection of Coconut Oil in Butter Fat by Means of Polenske's Distillation Method and Bömer's Phytosteryl Acetate Test. C. Barthel and K. Sondén. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1914, **27**, 439-453.)—Numerous samples of Swedish butter examined had Reichert-Meissl values of from 25·7 to 31·2, and in no case did the Polenske value exceed the limit laid down by Polenske for a corresponding Reichert-Meissl value. In most cases the addition of 5 per cent. of coconut oil to the butters could be detected by the method, whilst 10 per cent. could be detected with certainty. On the other hand, butters obtained from cows fed on coconut oil cake or beetroot leaves (not more than 2 kilos of the former or 40 kilos of the latter per animal per day) yielded Polenske values which exceeded the maximum figure for the corresponding Reichert-Meissl value, and on this account the method cannot be relied upon to detect the presence of added coconut oil in butter fat. Bömer's phytosteryl acetate test yields more trustworthy results. The cholesteryl acetate obtained from pure Swedish butters had a m.-pt. of 114·1° to 115·3° C.; in the case of butters from animals fed on coconut oil cake, the m.-pt. of the acetate was 115° to 115·9° C. The addition of 10 per cent. of coconut oil to the genuine butters increased the m.-pt. of the acetate from 116·2° to 117·1° C., but smaller quantities could not be detected with certainty. W. P. S.

Detection of Nickel in Fats. R. H. Kerr. (*J. Ind. and Eng. Chem.*, 1914, **6**, 207.)—In testing samples of cotton-seed oil, hydrogenated cotton-seed oil and mixtures of fats containing cotton-seed oil for nickel, a fugitive red colour sometimes appears after the addition of dimethylglyoxime and ammonia, closely resembling that obtained when a trace of nickel is present, except that it is fugitive and appears immediately after the addition of ammonia and never before. The fact that this reaction has been observed only with cotton-seed oil, and this taken together with the fact that this oil contains numerous basic organic compounds, renders it probable that the colour is due to some such body contained in the oil and extracted from it by the hot hydrochloric acid. It was found that if all organic matter in the acid extract be destroyed by evaporation with nitric acid before applying the test, samples previously showing the fugitive red coloration no longer did so, and it is recommended that this procedure be always adopted, as, in addition to other advantages, the residues so obtained are cleaner and more readily soluble in water. A larger sample of the fat may also be employed if desired. H. F. E. H.

Rancidity of Olive Oil and Oxidation of Oleic Acid in Presence of Sunlight. F. Canzoneri and G. Bianchini. (*Annali Chim. Appl.*, 1914, **1**, 24-32.)—Equal quantities of the same olive oil with an acidity of 1·35 per cent. were exposed to the air in sunlight and in the dark for fifteen days. In the first case the samples had become rancid, whilst in the second they were unaltered in appearance or composition. A sample of the oil was also sealed up in a test-tube and exposed to sunlight for fifteen days. The colour and composition remained unaltered. From these results the conclusion is drawn that rancidity is produced by oxidation promoted by light, and is not due to the action of micro-organisms. Oleic acid, after an exposure of five months to the action of air and light, contained various oxidation products, including formic, azelaic, and dihydroxystearic acids, a white compound melting at 50° to 51° C. (probably stearolactone), and the acids formed in the decomposition of oleic acid ozonide. Other experiments in which oleic acid was exposed for a week to the action of oxygen and light showed that products of a similar character were formed, as obtained by Molinari, by treating oleic acid with ozone. (*Cf.* ANALYST, 1906, **31**, 412.) C. A. M.

Use of Carbon Tetrachloride in the Extraction and Estimation of the Active Principles of Drugs and Medicinal Plants. G. Gori. (*Boll. Chim. Farm.*, 1913, **52**, 891; through *Chem. Zentralbl.*, 1914, I., 1377.)—For the estimation of caffeine in kola nut, a paste of 15 parts finely ground nut, 10 parts magnesium oxide, and water, is dried, powdered, and extracted for ten to twelve hours with carbon tetrachloride in a Soxhlet apparatus. The solvent is removed and the residue extracted by boiling with three successive quantities of water; the extract is filtered and evaporated to the point of crystallisation. Three determinations gave 1·61 to 1·77 per cent. caffeine, while three in which chloroform was used as solvent gave 1·60 to 1·78. In the estimation of green coffee-berries the dried mixture of substance and magnesium oxide was moistened before extraction: with carbon tetrachloride 1·19, and with chloroform 1·01, per cent. caffeine, were obtained. For the separation and estimation of caffeine and theobromine in fluid extract of kola nut, 20 grms. of sample are evaporated to half the original volume, worked into a dry powder with magnesium oxide, and extracted with carbon tetrachloride for twelve hours, whereby the caffeine is removed. The theobromine is then extracted with chloroform.

O. E. M.

Melting-Points of Constituents of Colophony. L. Paul. (*Chem. Rev. Fett- u. Harz-Ind.*, 1914, **21**, 102-105.)—American colophony contains a constituent which dissolves with difficulty in cold petroleum spirit, but is readily soluble in dilute sodium carbonate solution. On the addition of a mineral acid there separates a resin acid melting at 74° to 75° C., which constitutes about 80 per cent. of the colophony. When this acid is kept it absorbs moisture, and is gradually transformed into products of higher m.-pt. (103° to 105° C.), whilst other acids of still higher m.-pt. (*e.g.*, 130° to 135° C.) are also formed by hydration, and are present in variable proportions in the original colophony. The smaller portion of colophony which is readily soluble in petroleum spirit, but dissolves with difficulty in sodium carbonate solution, yields, on distillation, a resin acid crystallising in plates, and probably identical with γ-abietic acid. This also appears to be a hydration compound of the main acid of the colophony. The disintegration of colophony into powder is to be attributed to this hydration of its principal acid of m.-pt. 75° to 77° C. This also accounts for the bad weathering properties of colophony varnishes.

C. A. M.

Furfural in Cider Vinegar. A. A. Anderson. (*J. Ind. and Eng. Chem.*, 1914, **6**, 214.)—Out of twenty-eight samples of cider vinegar of known purity, nineteen gave a positive test for furfural by the aniline acetate test, and the presence of furfural cannot, therefore, be taken as an indication of the addition of wood acetic acid (*cf.* ANALYST, 1913, **38**, 565) or of caramel, when the latter is tested for by means of Fiehe's resorcinol reagent. H. F. E. H.

Refraction of Milk Serum (Ackermann's Method). R. Windisch. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1914, **27**, 466-469.)—Examination of the mixed milk of a herd of cows by Ackermann's method (ANALYST, 1907, **32**, 117) showed that the refraction of the serum generally ranged from 39 to 40 (refractometer degrees). The investigation extended over one year, and both morning and evening milks were examined separately. Only three samples gave a reading lower than 38. The milk from individual cows was also examined separately, the results obtained showing that the refraction of the serum tends to rise towards the end of the lactation period. At the beginning of the period the readings obtained lay between 38 and 39, whilst towards the end it was usually between 39 and 40. The highest reading obtained for a serum was 41·25 (*cf.* McCrae, ANALYST, 1914, 212). W. P. S.

Temperature Corrections to be applied to the Specific Gravities of Malt Worts and Beers. F. E. Day and S. L. Amos. (*J. Inst. of Brew.*, 1914, **20**, 196-198.)—It is often a convenience to determine the specific gravity of the worts obtained in the "Extract" determination of malt at temperatures other than the standard (60° F.). Since the coefficient of expansion of malt wort and water is not the same, it is not sufficient merely to take the ratio of wort to water at the desired temperature, and a further correction is necessary. It is found that between the limits of 40° F. and 70° F. for worts, whose specific gravities lie between 1024° and 1031° (water = 1000°), a correction of 0·019° is required to be added for every 1° F. that the solution is weighed above 60° F., the same amount being deducted for every 1° F. below 60° F. This correction applies only to specific gravities when determined in an ordinary Bohemian glass bottle, and is not to be used for solutions other than malt worts.

The necessary corrections to be applied to the apparent gravity of beer when using the hydrometer are as follows:

Hydrometer.	Range of Apparent Gravity. Brewer's Pounds.	Corrections in Fractions of One Brewer's Pound per Barrel.							
		45° F.	50° F.	55° F.	60° F.	65° F.	70° F.	75° F.	80° F.
		Deduct.			—	Add.			
Brass	2–5	0·3	0·2	0·1	—	0·15	0·35	0·50	—
,,	5–9	0·4	0·3	0·2	—	0·20	0·40	0·60	—
,,	9–15	0·5	0·35	0·25	—	0·25	0·45	0·70	—
Glass	2–5	0·35	0·25	0·10	—	0·20	0·40	0·60	0·85
,,	5–9	0·45	0·35	0·20	—	0·25	0·45	0·70	1·00
,,	9–15	0·55	0·40	0·25	—	0·30	0·50	0·80	1·10

H. F. E. H.

Estimation of Amino-Acid and Polypeptide-Nitrogen in Barley, Malt, and Beer, by Titration with Acid after Treatment with Formaldehyde. L. Adler. (*Zeitsch. ges. Brauw*, 1914, **37**, 105-133; through *J. Inst. of Brew.*, 1914, **20**, 291-294.)—Eighty c.c. of the extract of barley, malt, or other material, are measured into a 100 c.c. flask and heated in boiling water for ten minutes to precipitate the coagulable proteins. The cooled liquid is then treated with 1 to 2 grms. of solid barium chloride and 1 c.c. of phenolphthaleïn solution (0·5 grm. in 50 c.c. of 96 per cent. alcohol and 50 c.c. water). When the barium chloride has dissolved, a saturated solution of barium hydroxide is added until the solution becomes strongly red. The flask is filled to the mark and left for ten to fifteen minutes to allow of the quantitative separation of the phosphates present. The solution is filtered, and two portions (each of 40 c.c.) are rendered neutral to litmus ($p_H = 6{\cdot}8$) by addition of acid until a drop of the liquid placed on azolitmin paper (Kahlbaum) produces an immediate colour similar to that produced by a mixture of equal volumes of two solutions, one of which contains 9·078 grms. of KH_2PO_4, and the other 11·876 grms. of $Na_2HPO_4 2H_2O$ per litre. Equal volumes of these solutions should show a neutral tint on the azolitmin paper, while a mixture of 7 parts of the first with 3 parts of the second will show faintly acid, and 3 parts of the former with 7 parts of the latter should be faintly alkaline. The colour, which is liable to change, should be observed at once against a black background. Each of the 40 c.c. test portions is treated with 10 c.c. of 35 per cent. formaldehyde, recently neutralised by adding 0·2 c.c. of the 0·5 per cent. phenolphthaleïn solution (per 10 c.c.), and then $\frac{N}{2}$ caustic soda until a faint but distinct red is reached. As a blank test, 10 c.c. of the formaldehyde solution are diluted with 45 c.c. of distilled water, brought, if necessary, to the same tint as the real test solutions with Bismarck brown (0·592 per litre), and titrated with $\frac{N}{8}$ barium hydroxide until a strong red colour is attained ($p_H = 9·1$). This red should not be so strong that further addition of baryta produces no increase in intensity. The actual solutions to be analysed should next be titrated to exactly the same tint under the same conditions, it being remembered that the red colour of the phenolphthaleïn soon fades. The volume of $\frac{N}{8}$ barium hydroxide in c.c., less the volume required by the blank, gives, when multiplied by 2·8, the number of mgrms. of nitrogen determinable by "formalin titration" in the 40 c.c. portion of liquid. Ammonium salts react with formaldehyde, so that, if present, they should be removed by distillation, or estimated and allowed for.

Since proteolytic changes occur during the extraction of malt or barley with water even at 1° to 2° C., such material must first be boiled with alcohol for two hours to destroy the enzymes; the aqueous extract is then made after the removal of the alcohol. If this is not done, values two or three times too high are obtained. About 90 mgrms. of "formalin" nitrogen were found in 100 grms. of pale malt, 65 mgrms. in 100 grms. of dark kilned malt, and 17 mgrms. per 100 grs. in a sample of barley. Pale beer showed 175 mgrms. per litre (partly amino-acid and partly polypeptide in character). Since the colour of malt liquors renders exact titration very difficult, decolorisation with charcoal may have to be resorted to (*cf.* ANALYST, 1908, **33**, 19).

H. F. E. H.

Muta-rotation as a Source of Error in the Double Polarisation (Clerget-Herzfeld) Method of Determining Cane Sugar. V. Staněk. (*Zeitsch. Zuckerind. Böhm.*, 1914, **38**, 289-298.)—Muta-rotation is stated to account for the higher constants recently obtained by L. Steuerwald (ANALYST, 1914, 86), as compared with the values established by Herzfeld, now generally used. It was observed that the constant depends upon the time elapsing between completing the rapidly cooled inverted solution to the mark and making the polarimetric observation. When the interval was three to five minutes the values obtained were in accord with those established by Herzfeld—viz., 142·66 for 13 grms. of sucrose per 100 c.c. at 0° C.; whereas when it was fifteen to twenty minutes the constants were in agreement with those found by Steuerwald—viz., 143·00. After fifteen to twenty minutes equilibrium is reached, and in order to avoid error from muta-rotation, it is recommended by the author that the inverted solution should be rapidly cooled to normal temperature, and its volume completed to the mark, but that it should not be read until after the lapse of fifteen to twenty minutes. During this time treatment with purified animal charcoal or with a decolorising carbon may be made, if necessary. If this method of working be adopted, it would involve the use of the higher constant correct for the longer interval, and the value of 143·00 for the half normal sugar weight solution at 0° C. is proposed.

J. P. O.

Detection of Raw Beet Products in Admixture with Raw Cane Products. A. R. Ling. (*J. Inst. of Brew.*, 1914, **20**, 185-192.)—The author draws attention to the fact that the nitrogen content of cane sugar products is less than that of beet sugar products of the same cane sugar content, thus rendering it possible to detect the latter when mixed with cane sugar products. In the following figures, which are typical of many, "albumenoids" are calculated from nitrogen (not including nitrate or nitrite nitrogen) by the factor 6·25, and the cane sugar values in the molasses were determined by the Clerget process:

RAW PRODUCTS.

	Cane Sugar. (Polarisation.)	Albumenoids.
	Per Cent.	
ane : Mauritius	85·0	0·48
"	81·0	0·85
Penang	88·0	0·39
Jaggery	75·0	0·59
Demerara	95·0	0·47
Cuba	96·0	0·41
"	84·0	1·14
Beet : First runnings	94·6	1·30
" "	95·6	0·89
Second runnings	92·3	1·77
" "	91·2	2·19

	Cane Sugar. (Clerget.)	Albumenoids.
	Per Cent.	
Molasses (cane), four samples	55·0 to 58·0	3·00 to 4·49
" (beet), three samples	48·0 to 49·5	9·57 to 10·55

For commercial invert sugars purporting to be made from cane only, it is suggested that the following limits for albumenoids should be adopted: No. 1 invert 0·3 per cent.; No. 2, 0·5 per cent.; and No. 3, 0·75 per cent. In cases where these limits are somewhat exceeded, additional evidence (odour and flavour) should be adduced before coming to a definite conclusion that beet sugar is present. There is evidence to show that a considerably larger proportion of the nitrogenous substances found in cane products is removed by charcoal than is the case with beet sugar, a fact which can be explained when it is remembered that the organic nitrogenous substances in beet are of much lower molecular weight than those met with in cane sugar, these last more nearly approximating to the true proteins. No importance can be attached to the composition of the ash as a factor of diagnostic value in determining whether the origin of a commercial sugar be cane or beetroot.

H. F. E. H.

Estimation of Sulphate in Honey and Other Substances Rich in Sugar. F. E. Nottbohm and H. Weinhausen. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1914, **27**, 581-587.)—Substances such as honey, fruit syrups, wines, etc., should not be incinerated over a gas burner when it is desired to estimate the total sulphate in the ash, as considerable quantities of sulphur compounds are absorbed from the combustion products of the gas; too high results are also obtained when the substance is ignited in a silica muffle, and, in either case, the error is largely increased when the substance is treated with alkali previous to the ignition. On the other hand, ignition over a spirit flame is not to be recommended, as a portion of the sulphate present is reduced by the large amount of carbonised matter, and loss of sulphur compounds takes place. The best method consists in treating 20 grms. of the sample with 0·2 grm. of magnesium carbonate and 50 c.c. of nitric acid of sp. gr. 1·4; when the first violent reaction is over, the mixture is heated over a spirit flame, and further quantities of nitric acid are added until nitrous vapours cease to be evolved. The solution is then evaporated with hydrochloric acid, and the resulting sulphuric acid is estimated in the usual way. W. P. S.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Carbon Dioxide and Plants. R. Klein and E. Reinau. (*Chem. Zeit.*, 1914, **38**, 545-547.)—Experiments with various plants (*aspidistra, philodendron, nephrolepsis, pteris, and begonia*) showed that carbon dioxide is very beneficial to their growth. Plants grown in an atmosphere containing from 3·5 to 4·5 parts of carbon dioxide per 1,000 increased in growth by from 124 to 238 per cent. as compared with a similar series of plants grown in the ordinary atmosphere, when the experiments were continued for a period of seven weeks. W. P. S.

Quantitative Extraction of Diastases from Plant Tissues. R. W. Thatcher and G. P. Koch. (*J. Amer. Chem. Soc.*, 1914, **36**, 759-770.)—In order to obtain a quantitative value for the diastatic activity of flour, the authors assume that the enzyme must first be extracted in aqueous solution, with the result that some fraction of the enzyme is "used up" owing to its action on the flour starch during the extraction period. In order to obviate this they recommend extraction at 0° C., and give figures showing that when extraction of flour, bran, wheat, malt, etc., is carried on at this temperature for periods of one, three, and five hours practically similar results are obtained, which would not be the case were enzymic activity continuous during such period. Kjeldahl's law of proportionality (*Comptes rend. des travaux du lab. de Carlsberg*, 1879, **1**, 109-157) is assumed to hold for all these materials up to the production of 40 per cent. of maltose, the diastatic activity being expressed in terms of the maltose found per grm. of material employed when digestion of the diastase solution with soluble starch is carried on for thirty minutes at a temperature of 40° C. It is stated that a "quantitative extraction of the diastases present in vegetable tissues ground to the customary degree of fineness for ordinary analyses," is obtained by employing the method described.

H. F. E. H.

Note by Abstractor.—The authors have overlooked the fact first shown by J. L. Baker and H. F. E. Hulton (*J. Soc. Chem. Ind.*, 1908, **27**, 368) that the "Kjeldahl law of proportionality" does not strictly obtain up to a production of 40 per cent. of maltose in the case of flour. There is no reference to the work of Brown and Morris (*J. Chem. Soc.*, 1893, **63**, 604), and Ford and Guthrie (*J. Inst. Brewing*, 1908, **14**, 61), who showed that the diastatic activity of raw barley itself is greater than that of any extract made from it. The great influence that amphoteric substances have upon the quantitative production of maltose from starch when flour, barley, etc., are employed is ignored. H. F. E. H.

Method for Determining the Value of Disinfectants. C. A. Duyser and W. K. Lewis. (*J. Ind. and Eng. Chem.*, 1914, **6**, 198-200.)—Exception is taken to the Rideal-Walker and other methods generally employed for the following reasons: (1) The use of an excessive number of bacteria depletes the disinfecting solution before the culture is rendered sterile; (2) an unknown volume is withdrawn for testing, in that the standard loop may vary in its carrying capacity by as much as 80 per cent.; and (3) it is impossible to determine from the broth tube inoculated how complete the sterilisation was at the time of withdrawing the sample. The principle of a method obviating these objections is as follows: The disinfectant to be tested for its relative bactericidal power is diluted with water to three or four definite concentrations, depending on its strength. Pure phenol is similarly diluted. Into a series of these known concentrations of phenol and the disinfectant is placed an equal volume of a standard bacteria culture, and the mixture allowed to remain an exact number of minutes. An aliquot portion of each mixture is plated out and incubated until the colonies representing the surviving bacteria can be counted. At the same time plates of equal dilutions of the culture, but without the disinfectant, are incubated. The ratio of surviving organisms to the number present in the undisinfected plates is then plotted against the dilution, and two curves, one for the standard phenol and one for the disinfectant, are thus obtained. From these, the relative toxicity of the two disinfectants can be read off at any desired point on the curves, and a coefficient either for total sterilisation or any determined percentage of sterilisation may be calculated. Though desirable it is not necessary, as in other methods, that the most concentrated dilution of the disinfectant used should show total sterilisation, for if at least three points are found to lie fairly well on a smooth curve, interpolation for the point of complete sterilisation is permissible. H. F. E. H.

Composition of the Soft Roe and Hard Roe of Fishes. J. König and J. Grossfeld. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1914, **27**, 502-520.)—Soft roes obtained from herrings and carp had the following general composition: Water, 75·62 to 78·47; total nitrogen, 3·07 to 4·12; fat, 3·17 to 4·42; ash, 2·21 to 2·34 per cent. The total nitrogen consisted of nitrogen in the form of albumin, 0·04 to 0·06; as "roe substance," 1·76 to 3·49; and as bases and amino-acids, 0·57 to 1·17 per cent. Xanthine, creatinine, free amino-acids, and "protamines" combined with nuclein substances, were present. Hard roes from carp, herrings, pike, cod, etc., consisted of—Water, 63·53 to 73·98; total nitrogen, 3·55 to 4·50; fat, 1·40 to 4·19; ash, 1·24 to 2·13 per cent. The quantity of albumin present varied from 0·15 to 4·83 per cent., and of ichthulin from 11·47 to 24·33 per cent. Three samples of caviar examined contained—Water, 46·06 to 59·39; total nitrogen, 3·53 to 4·18; fat, 4·44 to 16·12; ash, 6·21 to 9·81; sodium chloride, 5·36 to 8·36 per cent. The hard roes and caviars also contained tyrosine, histidine, arginine, lysine, xanthine, and amino-acids. Nucleins were also present. Whilst the hard roes contained from 0·19 to 0·43 per cent. of free lactic acid and from 0·20 to 0·27 per cent. of oleic acid, the caviars contained up to 1·38 per cent. of lactic acid and 2·82 per cent. of oleic acid.

W. P. S.

Estimation of Hæmatin in Blood. A. Azadian. (*Bull. Soc. Chim. Belg.*, 1914, **28**, 91-94.)—The detection of hæmatin plays an important part in the examination of suspected bloodstains. According to Strzyzowski's method, a trace of the substance is placed on a microscope slide, moistened with a mixture of 1 c.c. each of alcohol, water, and glacial acetic acid, containing 2 drops of hydriodic acid, covered with a slip, and heated to boiling for some seconds, the loss being made up with the mixture indicated. On cooling, the hæmatin crystallises in the form of elongated rhombohedral tablets, isolated or in groups. Distinct crystals can be obtained with 0·005 mgrm. of fresh human blood. The author has estimated the quantity of hæmatin by Strzyzowski's method in the freshly drawn blood of various animals. The blood was immediately defibrinated and strained through a fine sieve; very rapid work is necessary, and the estimation should be effected within two hours after drawing the blood. Five c.c. of the defibrinated blood are added rapidly, in small portions at a time, to 5 c.c. of glacial acetic acid. Immediately afterwards 5 c.c. are added of a mixture of 25 c.c. of alcohol and 1·5 c.c. of colourless hydriodic acid (sp. gr. 1·5); the whole is shaken vigorously, and boiled for about two minutes. Fifteen c.c. of boiling water are at once added, and the tube is centrifuged for ten minutes at 2,000 revolutions per minute. The water is poured off, the crystals are washed three times with water and twice with alcohol, dried in the oven, and weighed. The quantities of hæmatin obtained per litre of defibrinated blood varied considerably with different animals—*e.g.*, mule, 7·70 grms.; horse, 6·48; ox, 6·0; calf, 3·72; sheep, 3·28; goat, 1·45. Variations were observed with different individuals of the same species, and it would be desirable to count the number of corpuscles in the blood in order to obtain comparative results.

J. F. B.

Quantitative Estimation of the Proteins Soluble in Salt Solutions in Wheat Flour. G. A. Olson. (*J. Ind. and Eng. Chem.*, 1914, **6**, 211-214.)—The author has already shown (Analyst, 1914, 33) that 50 to 60 per cent. alcohol extracts, in addition to gliadin, other proteins from flour. One per cent. sodium chloride extracts from flour, in addition to edestin, leucosin, and amino-bodies, gliadin amounting to about 30 per cent. of the total proteins of the flour; while 10 per cent. salt solution only extracts gliadin to the extent of about 5 per cent. of the total proteins. The gliadin nitrogen extracted by solutions containing sodium chloride can be determined and corrected for in making edestin and leucosin nitrogen determinations. It is found that the amount of nitrogenous substances extracted with salt solutions and directly coagulable by heat varies with the concentration of the solvent, the 10 per cent. concentration yielding higher results than the 1 per cent. Nitrogenous substances soluble in salt solution are partly or wholly soluble in dilute alcohol, varying with the concentration of the sodium chloride used. Detailed directions are given in the paper for carrying out the determinations of amide, globulin, albumin, and gliadin nitrogen in flour, based upon the above principles.

H. F. E. H.

"Tricresol" as a Substitute for Toluene in Enzyme Work. S. A. Graves and P. A. Kober. (*J. Amer. Chem. Soc.*, 1914, **36**, 751-758.)—It is concluded that tricresol (Schering) is preferable to toluene because it is a stronger germicide, is less volatile (b.-pt. 190° C.), is soluble in water to the extent of 2 per cent. without causing cloudiness and so interfering with optical work, and runs clearly from the pipette without clinging to the glass in oily drops like toluene. In 0·5 per cent. concentration it is recommended for use with trypsin, pepsin, urease, and diastase; while for erepsin and invertase it should be used in 0·2 per cent. solution, as with long standing in 0·5 per cent. concentration there is some retardation in the activity of these enzymes. With lipase the evidence is contradictory; 0·2 per cent. tricresol retards the action of castor-bean lipase on ethyl butyrate and triacetin, but accelerates the action of pancreatin on ethyl butyrate, and retards its action on triacetin. It is probable that tricresol will be useful as a preservative of urine. H. F. E. H.

Estimation of Allantoin in Urine. H. Hondovsky. (*Zeitsch. Physiol. Chem.*, 1914, **90**, 211-221.)—The urine is diluted so that it contains 1 per cent. of urea, neutralised if alkaline, and then treated with 1 per cent. sulphuric acid and 3 c.c. glacial acetic acid for each day's discharge. The urine is then precipitated with sufficient phosphotungstic acid, filtered, and the excess of phosphotungstic acid and sulphuric acid removed by means of lead oxide. If the filtrate from this treatment contains chlorides they are removed by precipitation with acetic acid and silver acetate. The filtrate is then treated with hydrogen sulphide to remove silver and lead, and the excess of hydrogen sulphide removed. The filtrate is neutralised with calcium carbonate, and absorbed carbon dioxide expelled. This liquid is then treated with a known volume of a solution containing 0·5 per cent. mercuric acetate, and 20 per cent. sodium acetate, and the whole made up to a known volume. This reagent precipitates the allantoin, which is estimated by titrating the excess of mercury with thiocyanate. After standing for about half an hour, and insuring that an excess of the precipitant has been used, the liquid is filtered and a measured portion of the filtrate treated with 10 c.c. of a ferric ammonium alum solution, and dilute sulphuric acid added until the red liquid possesses only a faint green shimmer. (Separation of calcium sulphate at this stage does not interfere with the subsequent titration.) The liquid is then titrated with a $\frac{N}{10}$ ammonium thiocyanate solution until the appearance of a yellow colour, which becomes more intense after addition of a further 1 to 2 drops. 0·00436 × number of c.c. $\frac{N}{10}$ ammonium thiocyanate solution = weight of allantoin in grms. E. W.

Violet Sodium-Nitro-Prusside Reaction of Urine. H. Yanagawa. *Biochem. Zeitsch.*, 1914, **61**, 256-281.)—This reaction discovered by Arnold (ANALYST, 1907, **32**, 94) is not given by urine during fasting, but occurs after taking food of the most varied character. The reaction is strong after the eating of meat foods and weak after other foods. The reaction is not given by any known constituent of urine, but is due to a substance which is precipitated by mercuric chloride and alkali, mercuric nitrate, and an ethereal solution of oxalic acid. The substance is probably a thioamide, which on boiling with acid or alkali decomposes, yielding a thiocyanate or hydrogen sulphide. E. W.

ORGANIC ANALYSIS.

Melting-Point of Asphalt. L. Schanin. (*Zeitsch. angew. Chem.*, 1914, **27**, 215-216.)—In specifying the melting-point of pitch or asphalt it is very necessary to state what is understood by the "melting-point," and by what method it is determined. Two methods are largely employed, both depending on liquefaction of a body of pitch sufficiently to allow a globule of mercury to sink through the mass. In Lunge's method the pitch is ground up and placed in a tube bent and drawn out to a point at the end. A drop of mercury is placed on the top, and the tube heated in water until the mercury sinks to the bottom. In Kraemer and Sarnow's method an open-ended tube is used, 6 to 7 mm. in bore, and a layer of molten pitch is solidified in the bottom end to a certain height. When cold, 5 grms. of mercury are placed on the top of the pitch, and the tube is heated in a water-bath. Considerable differences are found between the results of the two methods, and the results depend greatly on small details of manipulation. The author has devised an automatic apparatus based somewhat on the latter principle. In order to avoid discontinuity in the mass of pitch, the tube in which it is contained is drawn out to a capillary at the end, 1 mm. in diameter, for a distance of 10 mm. In this narrow tube the layer of pitch is allowed to rise to a height of 5 mm., and remains perfectly homogeneous; mercury is introduced above the pitch to a height of another 5 mm. The tube is fixed in a wide test-tube, side by side with a maximum thermometer, and the test-tube itself is fixed in a flask containing the liquid to be heated. Immediately beneath the tube containing the pitch are two metal plates, inclined towards one another, separated only by a distance of 1 mm. at the lower end. When the apparatus is heated and the pitch melts, the mercury falls between the two plates, thus closing an electric circuit, which operates a bell, and at the same time a pair of magnets. The magnets cut off the gas-supply and admit cold water or cold air to the apparatus. Thus the temperature recorded on the maximum thermometer indicates the melting-point of the pitch. J. F. B.

Colour Reaction for the Detection of Methyl Alcohol. C. D. Manzoff. (*Zeitsch. Untersuch. Nahr Genussm.*, 1914, **27**, 469-470.)—The test depends on the coloration which is obtained when nitro-methane is heated with ammonia and vanillin. A mixture of nitro-methane, ammonia, and vanillin is colourless when cold, but on heating develops a red colour, which disappears as the mixture is cooled. Nitro-ethane, under similar conditions, yields a faint yellow coloration. The test will detect the presence of 1 part of nitro-methane in 100,000 parts of nitro-ethane, and may be used for the detection of methyl alcohol in ethyl alcohol. Two hundred c.c. of the spirit are treated with 5 c.c. of concentrated phosphoric acid and distilled; the first 10 c.c. of distillate are then mixed with 5 grms. of red phosphorus and 20 grms. of powdered iodine, and heated for twenty minutes under a reflux apparatus. The mixture is now distilled, the distillate mixed with 3 grms. of silver nitrite and again distilled. The first 5 drops of distillate are treated with 5 drops of ammonia and 0·01 grm. of vanillin, and heated; a red coloration denotes the presence of methyl alcohol in the original spirit. The intensity of the coloration depends to a certain extent on the proportion of vanillin employed. Experiments with aromatic and aliphatic aldehydes and ketones, in place of vanillin, yielded negative results, except in the case of *p*-hydroxybenzaldehyde, which gave an orange-red coloration.

W. P. S.

Estimation of Methoxyl in Compounds containing Sulphur. A. Kirpal and T. Bühn. (*Ber.*, 1914, **47**, 1084-1087.)—The estimation of methoxyl in the presence of sulphur compounds by Zeisel's method is extremely unsatisfactory on account of the formation of silver sulphide in the absorption-flasks. The authors have devised a different method for the absorption and estimation of the methyl iodide, based on the fact that this body combines with pyridine, forming pyridine-methyl iodide, which latter may be titrated with silver solution, using sodium chromate as indicator. This method is entirely independent of the presence of sulphur compounds, and, further, is strongly recommended in the case of ordinary methoxyl estimations as being much simpler and just as accurate as the gravimetric method. For the decomposition of the substance to be analysed, the addition of acetic anhydride, as recommended by Goldschmidt, is of great advantage. In carrying out the estimation the authors distil in an atmosphere of hydrogen instead of carbon dioxide. The absorption vessels consist of two thick-walled tubes, each charged with 3 to 4 c.c. of pyridine, and, at the end, a small flask containing water to absorb any pyridine vapours carried over. When the reaction has started, a yellow coloration appears in the first absorption vessel, and it is generally sufficient to continue the distillation for an hour after this coloration appears. The apparatus is cooled in a current of hydrogen; the contents of the absorption-tubes are transferred to a glass dish and the aqueous pyridine solution is evaporated to dryness on the water-bath. The crystalline residue is dissolved in water and titrated with silver solution in the ordinary manner until the red tint is permanent. By this method also the secondary reactions observed in the analysis of methylimides do not interfere with the accuracy of the results.

J. F. B.

Composition of Paint Vapours. H. A. Gardner. (*J. Ind. and Eng. Chem.*, 1914, **6**, 91-95.)—When linseed oil or similar drying oils are spread in thin layers, the absorption of oxygen which takes place is accompanied by the evolution of considerable quantities of carbon dioxide and organic substances; carbon monoxide is also evolved in small amount. Oil paints, however, containing lead or zinc pigments do not give off volatile metallic compounds. During drying, paints evolve acid substances soluble in water, such as formic acid, as well as substances of a fatty nature; carbon monoxide and dioxide are also present in the vapours. The type of pigment may directly affect the amount and character of the volatile substances, and the production of the latter is apparently stimulated by the presence of basic pigments. Aldehydic substances are also present in paint vapours, and probably have an effect on pathogenic bacteria; this would account for the sanitary value ascribed to oil-pigment paints. W. P. S.

Analysis of Petroleum Oil and Paraffin Wax. H. Kantorowicz. (*Chem. Zeit.*, 1914, **38**, 494-495.)—The suitability of hot ethyl acetate as a precipitant of asphaltic substances (ANALYST, 1914, 43) is maintained against the criticisms of Holde and Meyerheim (ANALYST, 1914, 178). The fact that when boiling alcohol was used to extract a mixture of pure ceresin with sand (and alcohol) 91·5 per cent. of the wax was recovered does not prove, as claimed by Holde, that the residue consisted of "soft asphaltum" only, since some ceresin was probably retained by the asphaltum. Nor has any proof been given by Holde that the substance precipitated by alcohol-ether is really "soft asphaltum." On the contrary, the author's experiments have shown that the true soft asphaltum of petroleum pitch is largely soluble in alcohol-ether, whilst, on the other hand, the excess of "asphaltum" found by Holde is a light-coloured product apparently distinct from asphaltum. The use of *n*-hexane is preferable to *n*-pentane or petroleum spirit, as proposed by Holde as a substitute for "normal benzine"; but all these substitutes act like benzine in precipitating ceresin, and are only suitable for use at relatively low temperatures. In reply to Holde's criticism that ethyl acetate precipitates less asphaltum than other precipitating agents, the author replies that his analyses (ANALYST, 1914, 44) show that it sometimes precipitates more and sometimes less than "normal benzine"; and hence he has distinctly advocated its use, not as a substitute for benzine, but as an additional reagent. Speaking generally, commercial oils may be graded more sharply by means of the ethyl acetate test than by the "normal benzine" test.

C. A. M.

Determination of Phenol in the Presence of Hexamethylenetetramine and Formaldehyde. L. V. Redman, A. J. Weith, and F. P. Brock. (*J. Ind. and Eng. Chem.*, 1914, **6**, 205-206.)—Phenol in the presence of hexamethylenetetramine may be accurately determined by the method already described by the authors (ANALYST, 1912, **37**, 515; 1913, **38**, 335). The presence of formaldehyde, however, seriously interferes with the phenol determination. It was found that the addition of strong ammonia to the phenol-formaldehyde mixture forms, with the aldehyde, hexamethylenetetramine, or some intermediate ammonia-aldehyde product which no longer interferes with the quantitative determination of phenol. Ammonia of 2 N strength was found satisfactory for the phenol-formaldehyde mixture, after which addition a stand of five minutes or longer is advisable. In determining phenol in presence of hexamethylenetetramine, the bleaching of the starch iodide colour by the thiosulphate is slightly retarded, and it is necessary to give a few seconds after the addition of the thiosulphate to allow the blue colour to disappear.

H. F. E. H.

Picric Acid as a Standardising Substance for Iodimetric and Acidimetric Titrations. A. Sander. (*Zeitsch. angew. Chem.*, 1914, **27**, 192-194.)—Picric acid possesses several advantages over other substances commonly employed for standardising volumetric solutions. It contains no water of crystallisation, is very stable, non-hygroscopic, and is obtainable in a very pure form. When any doubt of its purity exists, it may be checked by the melting-point and a simple gravimetric estimation by precipitation with nitron. A dilute solution of picric acid, slightly acidified by sulphuric acid, is heated to boiling, and 10 c.c. of nitron solution (1 : 10) in 5 per cent. acetic acid are added slowly. After cooling, the precipitate is collected in a Gooch crucible, washed with water, and dried at 110° C. The factor for picric acid is $\frac{229}{541}$ of the dried precipitate. Owing to the sparing solubility of picric acid in water, $\frac{N}{10}$ solutions cannot be prepared; $\frac{N}{25}$ and $\frac{N}{100}$ solutions are generally used. In employing picric acid for standardising purposes, a solution containing 40 grms. of potassium iodide and 10 grms. of potassium iodate is required; this need not be rigidly accurate. The quantity of iodine liberated from an excess of this solution by the picric acid is titrated by the thiosulphate solution to be standardised, the results being extremely sharp and accurate. Standard solutions of acids and alkalis are adjusted by the same reaction, using the thiosulphate solution standardised by picric acid. The author strongly recommends the iodimetric method for acidimetric and alkalimetric work generally; it is particularly useful in the case of coloured liquids and when working by artificial light.

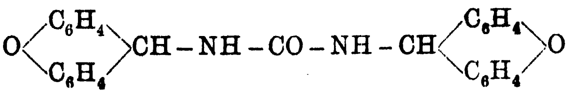
J. F. B.

Examination of Black Powder. W. O. Snelling and C. G. Storm. (*Zeitsch. Schiess- u. Sprengstoff.*, 1914, **9**, 21.)—The methods in use at the Bureau of Mines of the United States Government for the determination of the absolute densities, moisture, potassium and sodium nitrate, sulphur, and ash, are described.

O. E. M.

Tar Treatment of Roads. Revision of Road Board Specifications. (*J. Gas Lighting*, 1914, **125**, 807; through *J. Soc. Chem. Ind.*, 1914, **33**, 412.) —The chief modifications of the earlier specifications are as follows: *Tar No.* 1 (*for surface tarring of roads*).—The requirement that the distillate between 170° and 270° C. shall remain clear and free from crystals of naphthalene, etc., when kept at 30° C. for half an hour, may be waived in the case of tar supplied direct from gasworks, but it is preferable not to use tar containing much naphthalene. The tar should contain from 12 to 21 per cent. of "free carbon" (*i.e.*, residue left after extraction with benzene and carbon bisulphide). *Tar No.* 2 (*for making tar-macadam, and for surface tarring in very hot weather when the road crust is exceptionally dry*).—The tar should contain 12 to 22 per cent. of "free carbon," and may contain up to 25 per cent. by volume of tar (or distillates or pitch therefrom) produced in the manufacture of carburetted water-gas. *Pitch and Tar Oils.*—Prepared pitch from tar distilleries should contain 16 to 28 per cent. and commercial soft pitch 18 to 31 per cent. of "free carbon." The soft pitch and tar oils may contain up to 25 per cent. of products from carburetted water-gas tar.

Quantitative Gravimetric Estimation of Urea. R. Fosse. (*Comptes rend.*, **1914, 158,** 1076-1079.)—The formation of a sparingly soluble crystalline ureïn with xanthydrol has been utilised for the qualitative detection of urea in extremely dilute solutions. The derivative has the constitution—



$$O\left\langle\begin{matrix}C_6H_4\\C_6H_4\end{matrix}\right\rangle CH-NH-CO-NH-CH\left\langle\begin{matrix}C_6H_4\\C_6H_4\end{matrix}\right\rangle O$$

and its low solubility, together with its high molecular weight, seven times that of urea itself, afford an excellent means for the gravimetric estimation of urea by direct precipitation. Two methods of procedure are described: (1) The solution of urea, containing 1 grm. or more per litre, is first treated with 3·5 times its volume of glacial acetic acid, and then with 0·5 vol. of a 10 per cent. alcoholic solution of xanthydrol. After one hour the crystalline precipitate is collected, washed with alcohol, and dried. The results by this method show a normal error of about $-1·0$ to $-1·5$ per cent. for solutions containing 5 grms. of urea per litre, but with weaker solutions the error changes its sign and the results become too high. This is because the composition of the ureïn is affected by the large proportion of xanthydrol to urea in the reaction. Better results are obtained by method (2), adding the 0·5 vol. of 10 per cent. methyl alcoholic solution of xanthydrol in five separate portions at intervals of ten minutes, otherwise proceeding as before. The error by this method ranges from -1 to $-2·7$ per cent., the higher errors being found with the more concentrated solutions of urea. The gravimetric method possesses the advantage over methods depending on decomposition in the fact that the urea may at the same time be definitely characterised and the purity of the derivative confirmed by analysis. Schlœssing's method gives the most satisfactory results, the percentage of nitrogen being 6·66.

J. F. B.

INORGANIC ANALYSIS.

Convenient Standard End-Point in Titration of Alkali Carbonates with Hydrochloric Acid. G. P. Baxter. (*J. Amer. Chem. Soc.*, 1914, **36**, 656-657.)—Methyl orange is not altogether unaffected by carbon dioxide, and when the concentration of the latter is high, as in the titration of $\frac{N}{5}$ sodium carbonate with $\frac{N}{5}$ acid, the determination of the true end-point is attended with difficulty. Kuster (*Zeitsch. anorg. Chem.*, 1897, **13**, 141) suggested the use of a saturated solution of carbon dioxide, tinted with methyl orange, as control. This solution must be freshly preprepared, as the hydrion concentration is greatly depressed in time by the solution of a minute trace of alkali from the glass. The hydrion concentration of a solution of a monophosphate is but little affected by comparatively large additions of alkali, and the author recommends the use of suitable monophosphate solutions, tinted with methyl orange, as permanent standards. A solution of 15 grms. of KH_2PO_4 in

100 c.c. of water, tinted with methyl orange, exactly matches a saturated solution (about $\frac{N}{10}$) of carbon dioxide containing an equal amount of the indicator. Such a solution makes an excellent control solution for general purposes, as the solubility of carbon dioxide is such that solutions stronger than $\frac{N}{10}$ are seldom obtained, whilst the hydrion concentrations of solutions as weak as $\frac{N}{20}$ is but slightly less ($1{\cdot}2 \times 10^{-4}$ against $1{\cdot}7 \times 10^{-4}$), this difference being less than corresponds to 0·05 c.c. of $\frac{N}{10}$ hydrochloric acid in 100 c.c. A solution of 17·5 grms. $NaH_2PO_4.2H_2O$ in 100 c.c. water serves equally well, but this salt is less easy to prepare in a state of purity.

G. C. J.

Micro-Chemical Detection of Aluminium. E. Kratzmann. (*Pharm. Post.*, 1914, **47**, 101-102; 109-113; through *Chem. Zentralbl.*, 1914, I., 1459.)—On mixing a drop of a solution of an aluminium salt on an object glass with a drop of a reagent consisting of a mixture in equal parts of a 2 mol. solution of cæsium chloride and an 8 mol. solution of sulphuric acid, crystals of the cæsium alum of very characteristic form are obtained either immediately or after five to ten minutes, according to the concentration. The reaction is capable of detecting 0·3 μ grm. of aluminium nitrate or 1 μ grm. of ordinary alum. In applying the test to plant ashes one part of the powder is treated with a large drop of the reagent, whilst if much calcium carbonate is present it is advisable to add a little additional sulphuric acid. The method also gives good results in detecting aluminium in the sections of plants, the crystals being formed only in the places where it is present and not diffusing. In this way 130 plants belonging to different families have been examined, and aluminium has been found to be widely distributed in the vegetable kingdom. Certain plants, including *Lycopodium*, *Vitis*, *Symplocos*, *Oritis*, and *Anchusa* are particularly rich in aluminium. The so-called "alumina bodies," said to occur in the leaves of the *Symplocos* species could only be found in *S. polystachya* and *S. lanceolata*.

C. A. M.

Precipitation of Aluminium in Presence of Fluorides. H. Cavaignac. (*Comptes rend.*, 1914, **158**, 948.)—The quantity of alumina remaining unprecipitated from ammonium aluminium fluoride by ammonia was found to depend on the temperature and the quantity of ammonia in excess; it reached nearly 90 per cent. of the total when the excess of ammonia was small, and the temperature 100° C.

O. E. M.

Quantitative Separation of Arsenic from the Metals by Means of Hypophosphorous Acid. L. Brandt. (*Chem. Zeit.*, 1914, **38**, 461-463, 474.)—The author has recently described a modification of a method due to Engel and Bernard (*Comptes rend.*, 1896, **122**, 390) for the estimation of arsenic in iron, steel, and ores of iron and manganese. The method depends on the precipitation of the arsenic in the elementary condition by addition of sodium hypophosphite to the hydrochloric acid solution, with filtration of the precipitate and estimation of the arsenic by an iodimetric method. The author's modification consists in treating the precipitate, collected on an asbestos filter, with an iodate-iodide mixture. Owing to a trace of free iodine always present in such mixtures, the arsenic dissolves, slowly at first, but more and more rapidly, since iodine is one of the products of the series of reactions which follow the solution of the arsenic. The net result of these reactions is that each atom of arsenic sets free one atom of iodine, and the iodine thus liberated is titrated with thiosulphate (*Chem. Zeit.*, 1913, **37**, 1445, 1471, 1496).

The method has now been extended to the separation of arsenic from other metals besides those named. In the case of nickel, cobalt, zinc, chromium, tin, and bismuth, no difficulty is experienced in estimating arsenic, even when this is present in so small proportion that 10 grms. of the metal must be present in order to get a convenient quantity of arsenic to estimate. To the solution, which may contain hydrochloric or sulphuric but not nitric acid, and which should not exceed 120 c.c. in volume, 15 to 20 grms. of sodium hypophosphite are added, the mixture heated nearly to boiling, kept at this temperature for fifteen minutes after addition of 35 to 40 c.c. concentrated hydrochloric acid, and finally boiled for fifteen minutes. It is then filtered, and the washed precipitate is dissolved in iodate-iodide mixture as described. In the case of bismuth the quantity of hydrochloric acid should be greater (50 c.c.), and it should be added immediately after the hypophosphite, before heating to boiling.

Cadmium hypophosphite is somewhat difficultly soluble in hydrochloric acid, but arsenic may be separated from large quantities of cadmium if the precipitated arsenic is repeatedly treated with dilute hydrochloric acid to dissolve the cadmium hypophosphite which separates from concentrated solutions. With large quantities of aluminium the only difficulty is in getting the solution concentrated to 120 c.c. or less, but this difficulty does not arise if the aluminium is present as chloride. When metallic aluminium is dissolved in acids, arsenic is wholly eliminated as arsine, which can be oxidised to arsenic acid in an appropriate absorption apparatus containing bromine water; after elimination of the excess of bromine the estimation of the arsenic in this solution presents no difficulty.

Copper in amount exceeding 0·5 grm. interferes, and the separation of arsenic from even this small quantity of copper is only possible in hydrochloric acid solution. In sulphuric acid solution copper is itself reduced to metal by hypophosphorous acid. In hydrochloric acid solution this is not the case, but copper, if present in quantity, tends to contaminate the arsenic precipitate and to interfere with the quantitative precipitation of the arsenic, so that re-precipitation gives rise to low results. In presence of copper it is best to add some ferric salt, if none is present, and to precipitate the iron as hydroxide. This carries down all the arsenic. The precipitate is filtered off, washed once only as traces of copper do not interfere, dissolved in hydrochloric acid, and the solution treated with hypophosphite in the usual manner.

Arsenic can be separated from quantities of lead not exceeding 1 grm. in the manner above described, but for the estimation of very small proportions of arsenic in lead, involving the use of large quantities of lead, the latter must be separated as sulphate.

Experiments are described which show that the subsequent estimation of the metals, after separation of the arsenic by means of hypophosphorous acid, presents no difficulty.

G. C. J.

Standardisation of Volumetric Arsenite Solutions. E. Deiss. (*Chem. Zeit.*, 1914, **38**, 413.)—The standardisation of arsenite solutions with iodine is subject to an error which, in the author's experiments, reached 1 per cent. It may, however, be carried out accurately by means of a standard permanganate solution. A measured quantity of permanganate is run into a solution of 2 grms. of potassium iodide free from iodate in 200 c.c. water and 2 c.c. concentrated hydrochloric acid; 2 grms. of sodium bicarbonate are dissolved in the liquid, which is then nearly decolorised with the arsenite solution, and the titration completed with starch. Five grm. mols. of arsenic trioxide correspond to 4 of permanganate. O. E. M.

Detection of Bromine in Gases and Vapours. VII. Communication. I. Guareschi. (*Atti R. Accad. Scienze Torino*, 1914, **49**, 3; through *Chem. Zentralbl.*, 1914, I., 1373.)—In continuation of his work (ANALYST, 1913, **38**, 118, 518) on the detection of bromine by means of fuchsine and sulphurous acid, or Hofmann's violet decolorised with sulphur dioxide, the author points out that the copper-wire test of Beilstein does not distinguish between the halogens, and, like the modification of Vitali, fails with traces. In his apparatus a current of air is drawn successively through a flask containing the gas or liquid to be tested, a Mitscherlisch bulb-tube charged with chromic acid, a drying-tube, and a tube containing a paper moistened with the reagent. For organic compounds difficult to break down, the chromic acid must be warmed. The bromine in many aliphatic and aromatic compounds, and in samples of chlorine and iodine, was easily detected. Air containing 0·0004 grm. cyanogen bromide in 10 litres gives a sharp reaction; this substance is readily detected if present in fresh or sour milk, water, aqueous solutions of hæmoglobin or fresh urine, in the air which has been passed through them, but not in blood or albumin or stale urine, and with difficulty in solutions of meat extract. For the sensitiveness of the method with numerous organic compounds the original must be consulted. The reaction may be used in the detection of poisoning. Thus, bromine was found in the blood and in the aqueous extract of the blood-containing organs of an animal which had been anæsthetised with ethyl bromide or bromoform, and could be detected in the contents of the bladder after twenty-four hours. A mixture of 26 grms. of bullock's heart with 50 c.c. of water, a small quantity of tartaric acid, and 0·0082 grm. bromoform, and a mixture of 22 grms. of pig's liver with 150 c.c. water and 0·0015 grm. bromoform, gave sharp reactions. The failure of the reaction with cyanogen bromide in blood or in albumin is attributed to the formation of hydrocyanic acid and bromine, which forms a solid compound with the albumin.

O. E. M.

Estimation of Cobalt in Steel. P. Slawik. (*Chem. Zeit.*, 1914, **38**, 514.)—The following method depends on the fact that when iron is precipitated by zinc oxide, as in Volhard's method for manganese, but avoiding any unnecessary excess of the reagent, any cobalt (and nickel) present remains in solution, whereas chromium, vanadium, molybdenum, titanium, aluminium, copper, and silica are wholly precipitated. With tungsten steels it is unnecessary to separate the tungstic acid before treatment with zinc oxide, as the latter insures the complete precipitation of the tungsten. Cobalt is finally separated from nickel and manganese by precipitation with nitroso-β-naphthol in presence of so much hydrochloric acid that nickel is held in solution.

The drillings (2 grms.) are dissolved in dilute hydrochloric acid, and the solution is oxidised with potassium chlorate. Some steels—for example, steels of high tungsten content—may require the use of nitric acid, which should subsequently be expelled by evaporation to dryness, baking the residue, and dissolving it in hydrochloric acid. The excess of acid is removed by evaporation, and the solution transferred to a 500 c.c. flask, and diluted considerably. An emulsion of zinc oxide is added in small portions at a time with continuous shaking until the iron precipitate coagulates. The flask is then filled to the mark, and its contents shaken and filtered through a dry filter. Of the filtrate, 250 c.c. are acidified with hydrochloric acid, concentrated to about 100 c.c., and, after addition of 20 c.c. of concentrated hydrochloric acid, the cobalt is precipitated by the addition of 30 c.c. of a 2 per cent. alcoholic solution of nitroso-β-naphthol. After standing in a warm place with frequent agitation until the supernatant liquid becomes quite clear, the precipitate is filtered off, washed first with dilute hydrochloric acid, then with hot water, and is finally ashed with the paper, and the resulting Co_3O_4 is weighed. The ignited precipitate should always be tested for the presence of nickel, preferably by dissolving it in concentrated hydrochloric acid, adding tartaric acid, then ammonia to faint alkaline reaction, and, finally, dimethylglyoxime. Even with 25 per cent. nickel steels, the author obtained cobalt precipitates free from nickel, but the above test should never be omitted; it occupies little time, and has the advantage that if it discloses the presence of nickel this is obtained in a form in which it can be readily estimated and allowed for. G. C. J.

Separation of Iron and Manganese. O. Hackl. (*Jahrb. der K. K. Geolog. Reichsanstalt*, 1914, **63**, 151; through *Chem. Zentralbl.*, 1914, I., 1375.)—The solution containing the iron as ferrous sulphate and the manganese as sulphate, with sulphuric acid in excess, is neutralised, and boiled with 2·5 grms. potassium chlorate, 1·3 grms. zinc oxide, and a total quantity of 500 c.c. of water, for every 0·2 grm. of iron. Basic ferric sulphate is precipitated, and is washed with cold water; manganese remains in solution. The estimation is best carried out by volumetric methods.

O. E. M.

Investigation of the Methods of Analysis and Separation of Iron, Aluminium, Chromium, Zinc, and Manganese. G. Van Pelt. (*Bull. Soc. Chim. Belg.*, 1914, **28**, 101-127.)—Many of the methods employed for the estimation and separation of the above metals are based on the fact that the solutions of the salts (chlorides) of the trivalent elements (iron, aluminium, chromium) are strongly hydrolysed, whereas those of the divalent manganese and zinc are not. On this principle are based the methods of precipitation depending on the neutralisation of the hydrogen ion—*e.g.*, by barium carbonate, sodium acetate, or succinate. The author first estimated the degree of hydrolysis of solutions of the chlorides at a concentration of $N \times 10^{-3}$ by the diazoacetic ester method. From the values of K thus determined, it was calculated that solutions of ferric and aluminium chlorides are hydrolysed to the extent of about 80 per cent.; chromium, 40 per cent.; manganese, 10 per cent.; and zinc not at all. From this it follows that in the methods of separation based on the above principle the precipitates of the trivalent metals must always be contaminated with manganese, and this was confirmed by experiments. Chromium also, on account of its lower degree of hydrolysis, is precipitated far more slowly than iron and aluminium. Another set of quantitative separations in this group—viz., the separation of chromium and manganese, is based on oxidation, manganese being precipitated as Mn_3O_4 and chromium remaining in solution as chromate. The method described by Jannasch (ANALYST, 1912, **37**, 212) for this separation, using ammoniacal hydrogen peroxide, gives inaccurate results, owing to a portion of the chromium escaping oxidation and contaminating the manganese precipitate. But if a solution of the mixed chlorides be treated with ammonium persulphate and dilute nitric acid, the chromium is perfectly oxidised without precipitation, the manganese can then be separated quantitatively by precipitation with ammoniacal hydrogen peroxide. The separation of manganese from zinc by Jannasch's method is also defective, considerable quantities of zinc being included in the manganese precipitate. This may be avoided by adding to the ammoniacal peroxide solution an excess of ammonium chloride before running in the mixture of manganese and zinc salts, and satisfactory results can be obtained. By combining the two methods mentioned above, a good separation of the chlorides of manganese, chromium, and zinc may be obtained. The chromium is oxidised by acid ammonium persulphate, the manganese precipitated by ammoniacal peroxide in presence of ammonium chloride; the chromate is reduced by hydrogen peroxide in acid solution and precipitated by ammonia and ammonium chloride after evaporating off the peroxide, and the zinc is precipitated by sodium carbonate. J. F. B.

Colorimetric Estimation of Sulphur in Pig-Iron and Steels. G. Misson. (*J. Iron and Steel Inst.*, 1914, Advance Proof, p. 4.)—The method consists in getting the sulphur in the form of arsenious sulphide distributed evenly over some 50 sq. cm. of filter-paper. Filter-paper (Schleicher and Schüll, No. 598) is soaked in a solution containing 20 grms. of arsenious oxide and 60 c.c. of concentrated hydrochloric acid per litre. The paper is allowed to drain, but not to become dry. It is cut into pieces about 10 cm. square and preserved in a moist condition.

The apparatus employed consists of a number of slightly conical, flanged pots of stout glass, about 80 mm. in diameter and 95 to 100 mm. high; pieces of white felt, 10 cm. square and 5 mm. thick; pieces of ebonite, 10 cm. square and 1 to 2 mm. thick; and discs of lead weighing about 500 grs.

The sample (1 grm.) is placed in one of the pots, covered with 10 c.c. light petroleum (sp. gr. 0·71) and 50 c.c. hydrochloric acid (sp. gr. 1·4), and the pot is covered with a sheet of the arsenious paper, on which is superimposed a piece of felt, a square of ebonite, and a disc of lead. The felt insures a fairly even distribution of the escaping gases and correspondingly even distribution of the arsenious sulphide. About an hour is required as a rule. Comparison is made with stains obtained from steels, basic pig-irons, or Bessemer pig-irons of known sulphur content. The results are always within 10 per cent. of those yielded by more exact methods, and the method has been in use in the works of the Cockerill Company for more than four years. G. C. J.

Estimation of Small Quantities of Lead. M. Siegfried and W. Pozzi. (*Biochem. Zeitsch.*, 1914, **61**, 149-157.)—In the colorimetric estimation of lead as sulphide the presence of iron and the formation of lead sulphide which does not remain completely in colloidal suspension are sources of error. The authors find that iron does not interfere in the presence of $\frac{N}{1000}$ hydrochloric acid, and that gum arabic acts as a protective colloid and prevents the separation of lead sulphide. To 100 c.c. of the lead solution 9 c.c. $\frac{N}{100}$ hydrochloric acid and 1 c.c. of a 1 per cent. gum arabic solution are added, and to this mixture 10 c.c. of saturated hydrogen sulphide. A rough estimate of the lead content is made, and a similar solution containing a known and similar quantity of lead prepared. This second solution is used as a standard for the colorimetric estimation of the lead in the first solution.

E. W.

New and Sensitive Reaction of Lead. V. N. Ivanov. (*Chem. Zeit.*, 1914, **38**, 450.)—The reagent is a 2 per cent. solution of sodium hydrogen sulphite, which must be freshly prepared and give no acid reaction with methyl orange. When 50 c.c. of this solution is mixed with 50 c.c. of water containing much more than 1 part of lead in 1,000,000, an immediate opalescence appears, whilst the reaction is given, though not until after the expiration of some minutes, when 1 part of lead is present in 20,000,000 parts of water. Neither copper, silver, iron, aluminium, nickel, magnesium, nor calcium interfere, but barium and tin must be absent. In absence of other metals, normal sodium sulphite can be used instead of the acid sulphite. G. C. J.

Marshall's Manganese Reaction. E. Schowalter. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1914, **27**, 553-562.)—This reaction (ANALYST, 1901, **26**, 195) depends on the oxidation of manganese salts to permanganate by means of ammonium persulphate in the presence of silver salts, and yields satisfactory results when applied to the estimation of small amounts of manganese in water (*ibid.*, 1913, **38**, 477; 1914, 54). When dealing, however, with quantities of manganese exceeding a few mgrms. the method presents certain difficulties owing to the tendency of manganese dioxide to separate out during the oxidation. This objection may be overcome by means of the following modification of the method, and relatively large amounts of manganese may be estimated. The manganese salt (equivalent to not more than 0·2 grm. of manganese) is dissolved in water, 30 c.c. of nitric acid of sp. gr. 1·24, and 8 grms. of ammonium persulphate are added, and the mixture is diluted to 100 c.c.; this solution is then added, in small quantities at a time, to a solution consisting of 10 c.c. of $\frac{N}{10}$ silver nitrate solution, 15 c.c. of nitric acid, 10 grms. of ammonium persulphate, and 35 c.c. of water, the solution being maintained at a temperature of 50° C. during the addition. When all the manganese solution has been added the whole is kept at 50° C. for ten minutes, then cooled, and the permanganate is titrated with standard arsenious acid solution. With quantities of manganese of about 0·2 grm. the method yields 99·7 per cent. of the amount present. The reaction appears to be a catalytic one, in which silver peroxide acts as the catalyst.

W. P. S.

Electrolytic Estimation of Mercury in Fulminate. M. Losanitsch. (*Monatsh.*, 1914, **35**, 307.)—A solution of 0·4 to 0·5 grm. of the substance, dried at 80° C., in 2·5 to 3·5 c.c. of nitric acid of sp. gr. 1·40 is boiled to remove nitrous fumes, diluted to 120 c.c., and electrolysed in the cold; a Fischer gauze electrode is used with a current of 0·4 amp., falling to 0·25 amp. after fifteen minutes. At the end of six hours the electrode is washed with water, without interruption of the current, and with alcohol, and dried in an atmosphere of mercury vapour over fused potassium hydroxide. As a mean of six results, 70·39 per cent. was obtained; the theoretical percentage is 70·42. The use of a rotating gauze electrode and a current of 4·2 amps. allows the time to be reduced to twenty-five minutes.

O. E. M.

Composition of Monazite. (*Bull. Imp. Inst.*, 1914, **12**, 55-60.)—The following analyses of monazite from various sources are given:

Source.	ThO_2.	Ce_2O_3.	La_2O_3, etc.	Y_2O_3, etc.	Fe_2O_3.	Al_2O_3.	CaO.	SiO_2.	P_2O_5.
Ceylon	9·75	27·5	29·6	2·5	1·3	0·6	0·0	1·8	26·1
,,	9·49	27·2	29·6	3·9	0·9	0·2	0·5	2·7	26·1
,,	10·75	26·7	30·1	1·5	1·1	0·7	0·8	2·5	24·6
,,	10·29	27·4	30·1	2·1	0·8	0·2	0·4	1·0	27·7
,,	28·20	20·7	21·6	0·9	1·1	0·3	0·1	6·1	20·2
Travancore	10·22	31·9	28·0	0·5	1·5	0·2	0·2	0·9	26·8
,,	8·65	61·1		0·6	1·1	0·1	0·1	1·0	26·5
Malaya	3·40	33·7	32·5	0·9	0·7	0·0	0·3	1·5	26·6
,,	3·53	64·1		2·4	0·6	0·1	0·2	1·1	27·9
,,	9·41	60·0		2·8	1·1		0·3	2·2	23·7
Nyasaland	7·10	32·5	26·9	1·5	1·1	0·2	0·3	1·7	28·2
North Nigeria	5·00	30·7	30·0	2·7	3·0	0·4	0·2	1·2	26·3
,, ,,	3·20	36·5	30·0	0·4	1·2	0·1	0·2	0·6	28·3
,, ,,	8·00	30·5	28·8	1·4	0·8	0·2	0·2	1·8	28·2
South ,,	6·19	30·4	29·6	1·3	1·5	0·1	0·2	0·9	29·7
,, ,,	2·30	34·6	29·9	1·3	1·8	0·0	0·2	0·8	29·7
,, ,,	5·50	31·4	29·2	2·0	0·8	0·1	0·1	0·8	29·9
Brazil	6·06	62·1		0·8	1·0	0·1	0·2	0·8	28·5
,,	6·50	61·4		0·7	1·5	0·1	0·3	0·7	28·5
Virginia	14·23	29·9	26·7	—	—	—	—	2·8	26·1
North Carolina	6·49	31·4	30·9	—	—	—	—	1·4	29·3
Canada	12·60	24·8	26·4	4·8	1·1	—	1·5	0·9	26·9
Australia	1·23	36·6	30·2	—	—	3·1	—	3·2	25·1
Norway	9·34	28·1	29·6	1·8	0·7	0·2	0·5	1·7	28·3

The analyses of Ceylon and Nigerian monazite are selected from a large number made at the Imperial Institute, and it is stated that the average thorium content of Ceylon monazite appears to be about 10 per cent., and that few samples differ much from the average, whilst Nigerian monazite, though very variable in thorium contents, contains on the average nearly 6 per cent. G. C. J.

Colorimetric Method for Detecting and Estimating Nitrates and Nitrites. E. A. Letts and F. W. Rea. (*J. Chem. Soc.*, 1914, **105**, 1157-1161.)—Attempts to utilise the diphenylamine reaction for the colorimetric estimation of nitrates gave variable results, attributed to the non-uniform production of colourless diphenylbenzidine as an intermediate product. Perfectly satisfactory results were ultimately obtained by starting with the latter body as the original reagent instead of diphenylamine. A solution of 0·1 grm. of diphenylbenzidine in 50 c.c. of pure sulphuric acid is employed. 0·5 c.c. of the diluted nitrate solution is mixed in a porcelain crucible with 1·2 c.c. of sulphuric acid, and 0·3 c.c. of the diphenylbenzidine solution is stirred in. By preliminary tests the nitrate solution is diluted so that a deep sky-blue tint is obtained after ten minutes. The test proper is performed with six crucibles arranged in a tray of cold water, three being used for the standard solutions and three for the test solutions at different concentrations. The standard solution may contain 0·0005 mgrm. of nitric nitrogen per c.c. (0·361 grm. of potassium nitrate per litre, diluted 100 times). The standard crucibles may contain 0·1, 0·2, and 0·3 c.c. of the solution, in all cases made up to 0·5 c.c. by the addition of water. After adding the sulphuric acid an interval should be allowed for cooling before adding the reagent; the depth of colour is affected by any large variations of temperature and the time elapsing before observation. Nitrites may be estimated by oxidising with permanganate, and the total nitrate then estimated colorimetrically.

J. F. B.

Estimation of Nitrogen in Nitrates and Nitrocelluloses. A. Koehler, M. Marqueyrol, and H. Loriette. (*Ann. Chim. anal.*, 1914, **19**, 129.)—H. Pellet's criticisms (ANALYST, 1911, **36**, 513) of the nitrometer method described by M. Marqueyrol and D. Florentin (ANALYST, 1911, **36**, 243) are shown to be without foundation, and his modified Schloesing method (*loc. cit.*) is stated to be useless for the estimation of nitrates and of nitrocellulose, chiefly because it is impossible to correct for the solubility of nitric oxide in water, and because, in addition to nitric oxide, carbon dioxide and other gases are evolved in considerable quantities from nitrocellulose. The modified Devarda method (ANALYST, 1913, **38**, 171) and the nitrometer method give accurate results for nitrates and nitrocelluloses.

O. E. M.

Use of Sodium Citrate for Determination of Reverted Phosphoric Acid. A. W. Bosworth. (*J. Ind. and Eng. Chem.*, 1914, **6**, 227-228.)—The use of a so-called "neutral" solution of ammonium citrate for this purpose dates back to 1871. In view of the widely divergent results obtained by different chemists, attempts were made many years ago to frame directions that would insure the use by all workers of citrate solutions of approximately identical composition, and within the last year or two more than one satisfactory solution of this problem has at last been brought forward. But the author points out that uniformity in the composition of the citrate solution does not insure that uniformity in analytical results that is the ultimate desideratum. It is impracticable to conduct the digestions at 65° C. in stoppered flasks, and ammonia is always lost during the process, the loss depending not only on the character of the material extracted, but on the exact experimental conditions. The use of solutions of sodium citrate would be free from this objection, and might be expected to give results not widely different from those obtained with ammonium citrate, but more closely concordant among themselves. This has been tried, using a solution of sodium citrate containing 314 grms. crystallised sodium citrate ($C_6H_5O_6Na_3.5\frac{1}{2}H_2O$) per litre—that is to say, of the same molecular concentration as the ammonium citrate solution of sp. gr. 1·09 commonly employed. In every case the new solution dissolved less phosphate than did the ordinary ammonium citrate solution; but in most cases the differences were not large, and duplicate determinations with sodium citrate agreed far better than duplicate determinations with ammonium citrate. In three cases the new solvent extracted less than half as much phosphate as was dissolved by ammonium citrate, and in each of these cases bone was present.

The author does not suggest that the evidence brought forward is sufficient to justify the substitution of sodium citrate for ammonium citrate, but it is strong enough to invite attention. Should sodium citrate ever be adopted for this purpose, all trouble in securing a neutral solution would be eliminated, as the citric acid could be neutralised with sodium hydroxide, using phenolphthaleïn as indicator, or the neutral crystals of sodium citrate could be used. G. C. J.

Measurement of Radio-Activity of Springs. C. Engler, H. Sieveking, and A. Koenig. (*Chem. Zeit.*, 1914, **38**, 425-427, 446-450.)—The precautions necessary to secure accuracy in work of this kind are dealt with fully, and it is pointed out that, since the results are dependent to some extent on the shape and size of the ionisation chamber, they should always be reduced to absolute electrostatic units and corrected for the Duane effect (*Comptes rend.*, 1905, **140**, 581). Results obtained by different instruments are then comparable, since Duane's formula gives a closely approximate correction for the radiation absorbed by the walls of the ionisation chamber and for secondary radiation from these walls. The final result, being usually a very small fraction when expressed in absolute units, is most conveniently multiplied by 1,000 when it is given in Mache units. Instead of waiting three hours for the emanation and the products of its disintegration to attain a state of equilibrium, the authors prefer to proceed with their measurements with the least possible delay, noting the time (usually two minutes) which elapses between shaking the water with air and getting a reading. Even in two minutes the effect of Radium A and Radium C is notable, increasing the results about 19 per cent., and the authors make use of a table due to H. W. Schmidt (which they reproduce) for calculating—from their observed results and the time elapsing between shaking the water with air and their observation—the result which would have been obtained had it been possible to complete the experiment in an infinitely small space of time, before any part of the emanation had undergone further transformation. If results are all calculated back to this basis by Schmidt's table, then the final result so corrected and multiplied by 4×10^{-4} gives the emanation content of 1 litre of the water in microcuries.

An improved form of the author's "fontaktoscope" is figured, as in the orignal instrument (*cf.* Engler, *Zeitsch. Elektrochem.*, 1905, **11**, 714; Engler and Sieveking, *Zeitsch. anorg. Chem.*, 1907, **53**, 1), and the authors combat the view that the original instrument is inferior in accuracy to any other apparatus that has been devised for measuring the emanation content of waters. G. C. J.

Estimation of Silver in Collargol and Organic Fluids. P. W. Danckwortt. (*Arch. Pharm.*, 1914, **252**, 69-76.)—The total silver in collargol and similar preparations is estimated by igniting 1 grm. of the sample, and heating the ash with two successive portions of 5 c.c. of 25 per cent. nitric acid until nothing but silver chloride is left. This is dissolved in 15 c.c. of 10 per cent. ammonia solution added in three portions; the ammoniacal mixed solutions made up to 100 c.c., an excess of $\frac{N}{10}$ potassium cyanide solution added to an aliquot portion, and the liquid titrated with $\frac{N}{10}$ silver nitrate solution until silver iodide begins to separate. Results thus obtained are about 0·2 to 0·3 per cent. lower than those obtained gravimetrically. For the analysis of lymphs and preparations such as "argoferment," containing only traces of silver, $\frac{N}{50}$ solutions may be used for the titration. But if less than 1 mgrm. of silver is present a colorimetric process is more accurate. For this purpose the lymph is evaporated on the water-bath, and the residue gently ignited, with the addition, at the end, of small amounts of potassium nitrate. The white ash is heated twice with nitric acid and twice with ammonia solution as described, and the mixed solutions rendered slightly ammoniacal, diluted to 70 c.c. in a Hehner's Nesslerising tube, and treated with 10 c.c. of hydrogen sulphide solution. Since the lymph will contain some blood it is necessary to dissolve the iron sulphide by the addition of nitric acid. The colour due to the silver sulphide is then matched with that given under the same conditions by a solution of silver nitrate (0·1575 grm. per litre; 1 c.c. = 0·1 mgrm. of silver). Accurate results down to 0·02 mgrm. of silver are thus obtained, while chlorine or iron do not cause any error.

Commercial preparations of colloidal silver examined by the foregoing methods gave the following percentage results:

	Total Silver.	Silver as Chloride.		Total Silver.	Silver as Chloride.
Collargol I.	79·7	5·3	Argentum colloidale... (Siegfried)	78·5	1·6
„ II.	77·2	3·6			
Argentum colloidale ... (Gehe)	69·9	1·2	Lysargin (Kalle) ...	76·9	1·2
			Argoferment (Heyden)	0·041	trace
Argentum colloidale ... (Kahlbaum)	78·0	3·0			

C. A. M.

Estimation of Silver in Colloidal Silver. F. Lehmann. (*Arch. Pharm.*, 1914, **252**, 9-12.)—Many of the preparations of colloidal silver contain as much as 20 per cent. less silver than the original *collargol* of Heyden. For example, commercial samples were found to contain from 53 to 79 per cent. of silver. As silver chloride is an invariable impurity it is necessary to use a method of estimation which will bring it into solution. This may be readily effected by means of potassium permanganate and sulphuric acid. About 0·2 grm. of the colloidal preparation is shaken with 10 c.c. of water, and, after the addition of 10 c.c. of sulphuric acid, is treated little by little, with constant shaking, with 2 grms. of finely powdered potassium permanganate. After standing for fifteen minutes the mixture is heated until the upper part of the flask is washed clear of manganese dioxide, and is then cooled, diluted with 50 c.c. of water, treated with ferrous sulphate (to destroy excess of permanganate), and titrated with $\frac{N}{10}$ thiocyanate solution. C. A. M.

Estimation of Free Sulphurous Acid in Presence of Thiosulphate or Sulphuric Acid. A. Sander. (*Zeitsch. angew. Chem.*, 1914, **27**, 194-195.)—The iodimetric method of acidimetry may be used in combination with the ordinary iodimetric titration for various purposes. For the estimation of free sulphurous acid in presence of thiosulphate the solution is run into an excess of standard iodine, and the latter titrated back with standard thiosulphate; an excess of the iodide-iodate mixture is then added, and the free acid estimated by titrating the amount of iodine liberated. Similarly, with a mixture of sulphurous and sulphuric acids, the former may be estimated first by iodine, and the total acid then estimated by the addition of iodide-iodate solution. With the iodide-iodate solution free sulphurous acid alone behaves as a dibasic acid, whereas towards methyl orange it is only monobasic. Bisulphates can be titrated by the iodimetric method, showing accurately the half-combined sulphuric acid. J. F. B.

Estimation of Titanium as Phosphate. G. S. Jamieson and R. Wrenshall. (*J. Ind. and Eng. Chem.*, 1914, 6, 203-205.)—The method described is essentially that of Ericson (*Iron Age*, 1903, August 27, p. 4) for the determination of titanium in ferrotitanium and in ores. It is based on the precipitation of titanium phosphate in acid solution, after reducing iron to the ferrous state.

The solution, preferably containing from 10 to 30 mgrms. of titanium, is neutralised with ammonia if strongly acid, and then reacidified with 8 c.c. of concentrated hydrochloric acid and diluted to 100 c.c. Iron is next reduced by means of sulphur dioxide, or the reduction may be accomplished somewhat more quickly by means of hydrogen sulphide in the ammoniacal solution before reacidification. If the latter course be adopted the ferrous sulphide need not be removed by filtration, but is dissolved by the addition of 8 c.c. of hydrochloric acid and boiling. To the hot solution 20 c.c. of 10 per cent. ammonium phosphate solution are added, and the mixture boiled for thirty minutes. The precipitate is allowed to settle for an hour, and is then filtered off on a Gooch crucible, washed with hot water, and finally ignited over a full Bunsen flame and weighed as $TiPO_4$. Since the precipitate is gelatinous and tends to cake and then crack, in which condition it cannot be washed, this tendency must be checked by the use of only moderate suction and the addition of fresh wash-water before the precipitate can become compact. The gelatinous character of the precipitate also makes it inconvenient to work with quantities much larger than those recommended. With quantities such as these the error of the method never exceeds 0·4 mgrm.

With quantities of titanium less than 10 mgrms. the absolute error tends to increase, apparently because the precipitate fails to collect in filterable form; and with very small quantities the percentage error is very large indeed when proceeding as described above. By reducing the concentration of acid to about 3 c.c. per 100 c.c., and allowing the precipitate to settle for six hours, smaller amounts of titanium may be estimated with no greater absolute error than that stated above; but this modification of the method is not recommended for general use, as aluminium, if present, is partially co-precipitated with titanium when the concentration of hydrochloric acid is much less than 8 c.c. in 100 c.c. G. C. J.

Separation of Yttrium from the Yttrium Earths. Part I. H. C. Holden and C. James. (*J. Amer. Chem. Soc.*, 1914, **36**, 638-646.)—The authors have begun a systematic study of various methods for the separation of pure yttria from mixtures of this earth with oxides of erbium and other metals. Details of a number of fractional precipitations and fractional crystallisations are given. The methods investigated included fractional precipitation by means of hypophosphorous acid, sodium dimethyl phosphate, sodium cacodylate, ammonium phosphate, sodium monomethyl phosphate, sodium arsenate, sodium phosphite, chromic acid, and by means of potassium iodate, as well as fractional crystallisation of the bromates and fractional decomposition of the nitrates by careful fusion, followed by separating the soluble, unchanged nitrates from the basic salts and oxides by treatment with small quantities of water, the basic salts being sparingly soluble.

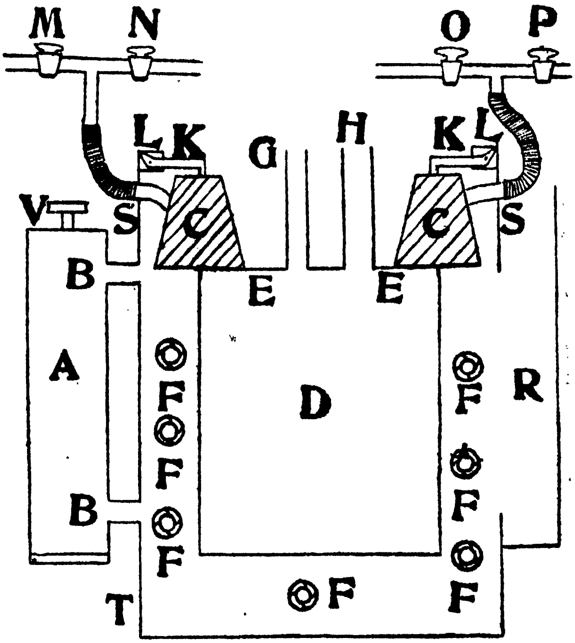
Of these methods, the chromate and phosphate separations are the most promising, the chromate and phosphate of yttrium being appreciably more soluble than the corresponding salts of the other metals. The precipitations by means of ammonium phosphate are conducted in hot 10 per cent. nitric acid solution. The chromate separation is conducted as follows: The mixed oxides are dissolved in chromic acid solution, the solution is neutralised by addition of excess of oxide, and is then diluted suitably and filtered. The filtrate is heated to boiling, and treated with 10 per cent. potassium chromate solution until a substantial precipitate results. This is filtered off and the filtrate treated with more potassium chromate, and so on, the final filtrate being treated with ammonia for the recovery of the purest yttria.

G. C. J.

APPARATUS, ETC.

Quantitative Micro-Elementary Analysis of Organic Substances by F. Pregl's Methods. J. V. Dubsky. (*Chem. Zeit.*, 1914, **38**, 505-506; 510-511.)—A description with seven illustrations, of Pregl's apparatus, for the exact determination of carbon, hydrogen, and nitrogen when only 5 mgrms. of substance are availabl (*cf.* Abderhalden's *Handbuch der Biochemischen Arbeitsmethoden*, 1912, **5**, II., 1307).

In view of the accuracy of the results, which is equal to that obtained by careful work with larger quantities, and to the rapidity with which combustions can be completed when using only a few milligrammes of substance, the author thinks these methods are likely to become the normal methods of combustion analysis. Certain special apparatus is necessary, such as a balance sensitive to 0·001 mgrm. with a load of 5 grms., but it is said that such balances can now be procured for 270 marks from W. H. F. Kuhlmann in Hamburg-Barmbeck, Steilhoperstrasse, 103. Among the pieces of apparatus figured are some absorption-tubes, which, it is said, do not perceptibly change in weight when left open on the balance for half an hour. These are straight tubes about 7 mm. in diameter, drawn out at either end to half that diameter. Each of these end-pieces is constricted to very small bore at two points, and the absorption-tube itself is similarly constricted about 1 cm. from each end. The refilling of such tubes with calcium chloride being out of the question, a copper block furnace is illustrated whereby the calcium chloride may be regenerated *in situ*, by connecting one end of the tube to a vacuum-pump and the other to a supply of dry air. G. C. J.



Adiabatic Calorimeter. **F. W. Gray.** (*J. Chem. Soc.*, 1914, **105**, 1010-1014.)—The author has devised a calorimeter with adiabatic mantle in which the heating and cooling are both effected by a stream of water in a flexible copper tube immersed in the mantle-water. The outer can *T* is connected by branches *BB* with a side-tube *A*, in which is situated a turbine stirrer. *R* is a tube fixed on to *T* opposite a slit, and serves to hold a Beckmann thermometer. To *T* are attached the blocks *L* and *L*, to which are hinged the levers *K* and *K*, movable downwards from the horizontal position, but not upwards. Three of these levers are arranged symmetrically. Pressing up against the levers are the corks *C*, against which the lid *E* of the submerged can *D* is pressed by the upward thrust of the mantle-water. The lid *E* is made watertight by vaseline. Access to the inside of *D* is obtained by means of tubes (*H* for a thermometer, and *G* for a stirrer); there is also a third tube not shown in the sectional illustration. A flexible copper tube *F* (0·63 cm. internal diameter and about 240 cm. long) is coiled around the submerged can, and soldered to inlet and outlet branches

SS; these are connected on one side to a supply of cold water (taps *P* and *N*), and on the other to a supply of water heated electrically to about 45° C. (taps *M* and *O*). By regulating these taps the temperature of the mantle-water may be controlled to follow closely the changes of temperature in the calorimeter. J. F. B.

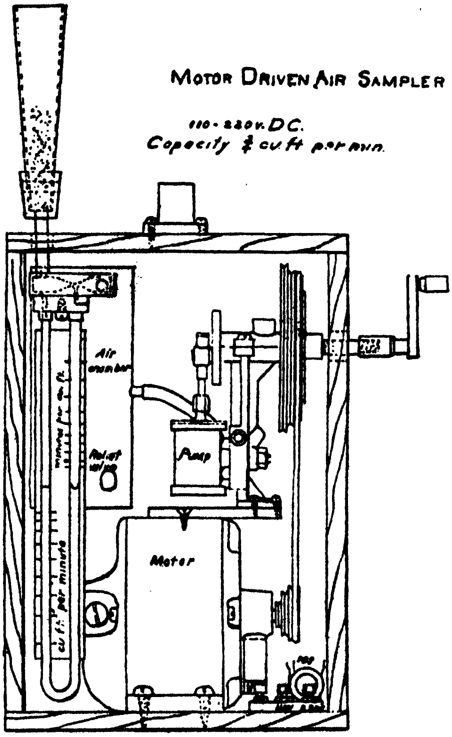
Apparatus and Method for Determining the Hardness of Butter Fat. A. E. Perkins. (*J. Ind. and Eng. Chem.*, 1914, **6**, 136-141.)—In the apparatus described a graduated rod, mounted in a frame and suitably weighted, is allowed to fall from a definite height on to the surface of the butter fat, the depth to which the rod penetrates the fat giving a measure of the hardness of the latter. The result may be expressed in the number of kilos required to displace 1 c.c. of fat at a given temperature. The fat is prepared for the experiment by melting it, stirring it while it cools until it is practically solid, and then maintaining it for twelve hours at a temperature not more than 1° C. from that at which the test is to be made; the temperature of the fat is adjusted accurately immediately before the determination. Most suitable temperatures for the tests lie between 0° C. and 15° C. The apparatus yields concordant results. W. P. S.

Two New Colorimeters. P. Verbeek. (*Zeitsch. angew. Chem.*, 1914, **27**, 203-208.)—Two new colorimeters are described and illustrated in detail. One of them is an immersion instrument, somewhat of the Dubosc type, but with more elaborate precautions to insure uniform illumination, and with one feature not embodied in any instrument of this type hitherto described. This consists in an arrangement whereby the light passes through an equal depth of the solvent in each half of the instrument, the shorter column of more concentrated coloured solution being superimposed on a column of the pure solvent; and as these liquids are separated by a glass plate the effect of this is neutralised by having a short column of the pure solvent in line with the less concentrated solution and separated from it by a similar glass plate. For very exact work this is said to constitute a notable improvement.

Immersion colorimeters being unsuitable for certain purposes—for example, for estimations depending on the development of a colour, the intensity of which is not proportional to the concentration of the substance to be estimated—another instrument has been designed, embodying the same principles for securing uniform illumination, but in which the liquids are viewed horizontally in cells of equal dimensions, the more concentrated liquid being diluted until an exact match is attained. For analytical purposes this is said to be the more generally useful instrument; but that first described is more convenient to manipulate, and in cases where intensity of colour is strictly proportional to concentration gives more accurate results. G. C. J.

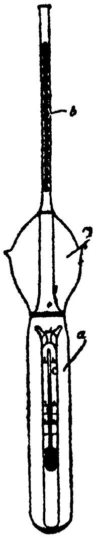
Double-Walled Crucible for Ignitions. A. C. Vournasos. (*Zeitsch. anal. Chem.*, 1914, **53**, 175-177.)—The outer wall of the apparatus is an ordinary crucible of any material suitable for the purpose in hand. The inner crucible for containing the substance is of special design, a flange extending horizontally from its upper edge for about 1 cm., and then being bent downwards for about 0·5 cm. This flange fits the top of the outer ordinary crucible like an ordinary crucible lid. An ordinary crucible lid may surmount the whole, but a special one is illustrated in the paper. The outer annulus of this lid rests flatly on the flange of the inner crucible, but the central portion is depressed to form a cylinder about 0·5 cm. high loosely fitting the inner crucible. From the centre of the lid rises a 0·5 cm. tube about 1 cm. high, ending in a T, also about 0·5 cm. in diameter. This T serves as handle and for the escape of gases—for example, in the coking of coal, or for the introduction of inert or reducing gas. The apparatus is made in porcelain and other materials by Warmbrunn, Quilitz and Co., Berlin, N.W., 40. G. C. J.

Apparatus for taking Dust and Bacteria Samples of Air. C. Baskerville. (*J. Ind. and Eng. Chem.*, 1914, **6**, 238.)—The apparatus includes an exhaust-pump of the friction type run by a $\frac{1}{32}$ horse-power motor, and a measuring apparatus placed between the pump and the sampling filter consisting of an air-chamber, the inlet of which is connected with the two ends of a U tube graduated on its distal arm. The whole apparatus is enclosed in an oak box 14 inches by 12 inches, air being drawn in through an opening at the top and forced out through an opening at the side. The upper opening is fitted with a small steel cup with a rubber stopper inside, having a hole large enough to fit the constricted end of the sugar or sand filter. At the base of the cup an extension passes through the wooden cover and screws on to the suction-chamber of the machine. The principle of the apparatus depends upon measuring the volume of the air sample by the time taken to draw it through, and when allowance is made for the resistance of the sand or sugar about three minutes per cubic foot is the rate of flow. H. F. E. H.



New Nephelometer and its Use in Analysis. F. Dienert. (*Comptes rend.*, 1914, **158**, 1117.)—The liquid under examination and a liquid of standard turbidity are placed together in a beam of light, and observed by means of a Dubosc colorimeter. The standard is altered by known amounts until the intensity of the light refracted by the suspended particles is the same for each liquid. By comparing in this manner the turbidities produced by the precipitation of barium sulphate, 1 mgrm.

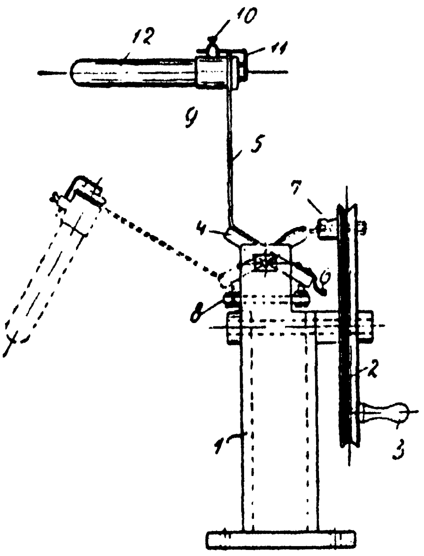
of sulphuric acid per litre can be determined in 20 c.c. of water. The sensitiveness is about the same for chlorine, calcium, and magnesium. O. E. M.



Pycnometer Spindle, a New Instrument for Use in the Estimation of Alcohol. H. Wüstenfeld and C. Foehr. (*Deutsch. Essigind.*, 1914, **18**, 114-116; 125-127; through *Chem. Zentralbl.*, 1914, I., 1537.)—The form of the apparatus is shown in the figure. The alcoholic solution to be tested is filled into the vessel *a* up to a definite point on the graduated stem *b*, and the apparatus is floated in toluene, the point to which it sinks being read on the graduated stem. *C* is a glass bulb which acts as a float. The volume of the solution in the apparatus is determined by weighing it when filled with water, and the weight of the solution is found from the toluene-displacement. The sp. gr. is then calculated from the ratio of weight to volume. The apparatus holds about 50 c.c. of solution. The following figures, showing the sp. gr. of toluene at different temperatures as compared with water at 15° C., may be found useful: 12·5°, 0·87228; 15°, 0·87005; 17·5°, 0·86774; 20°, 0·86550; 22·5°, 0·86314; 25°, 0·86089.

W. P. S.

Shaking Apparatus. O. Arendt. (*Chem. Zeit.*, 1914, **38**, 450.)—The apparatus figured is designed for shaking mixtures contained in test-tubes. The wheel, 2, adapted to be turned by hand or driven by power, carries a projection, 7, which at each revolution engages the projection, 6, attached to the wooden frame, 4. To this frame are attached stout wires, 5, ending in metal collars, 9, to which brass tubes, 12, large enough to accommodate a test-tube, are soldered. To the collars are also attached binding-screws, 10, which accommodate the stout bent wires, 11, which hold fast the corks closing the test-tubes. When the wheel is further rotated, the point, 6, is freed, and the frame carrying the test-tubes falls of its own weight into the position shown by the dotted lines, to be jerked up again at the next rotation of the wheel. G. C. J.



New Viscometer for General Scientific and Technical Purposes. E. C. Bingham. (*J. Ind. and Eng. Chem.*, 1914, **6**, 233-237.)—Bingham and White have already published a description (*Zeitsch. physik. Chem.*, 1912, **80**, 670) of a viscometer with which absolute viscosities can be measured with very great certainty. That form of apparatus is easily made, but since its dimensions must be accurately known for absolute measurements, the time consumed in calibration is considerable.

For general purposes it is preferable to calculate the absolute viscosities from measurements that are only relative. By this procedure the calibration is simplified and the apparatus may be made simpler and less delicate to handle. Many viscometers have been devised for relative measurements, but relative measurements are comparatively useless unless the results can be calculated to absolute units. It has been supposed that the relative measurements obtained by the use of instruments of the Ostwald type might be calculated to absolute units without difficulty. This is not generally true, and this is mainly due to the fact that the formula which seems to be almost universally used $\left(\frac{\eta}{\eta_0} = \frac{dt}{d_0 t_0}\right)$ takes no account of the loss of kinetic energy of the liquid within the capillary, this energy disappearing outside the capillary without helping to overcome viscous resistance within the capillary. Moreover, this correction does not come into the calculation in such a way that it can be made to disappear (*J. Chem. Soc.*, 1913, **103**, 959). Viscometers of the ordinary type are deficient because the pressure producing the flow through the capillary is not variable at will. The result is that with very fluid substances the kinetic energy correction becomes large unavoidably, and with rather viscous substances the time of flow becomes intolerably long, necessitating the use of several instruments. With a very long period of flow the difficulties due to clogging with dust particles become very great (Appleby, *J. Chem. Soc.*, 1910, **97**, 2,000). The necessity of the knowledge of the exact specific gravity of the liquid at each temperature where a viscosity measurement is desired lessens the convenience of this type of instrument. Both the untrustworthiness and the inconvenience of these instruments may be avoided by using variable pressure.

The instrument described and illustrated in this paper suffices for determining the viscosity of any liquid with an error not exceeding 0·1 per cent. For more accurate work absolute measurements must be made, as data do not exist for standardising a relative instrument for a much higher degree of precision. Detailed directions are given for the construction of the instrument, but this requires exceptional skill in glass-working, and the instrument may be purchased from Eimer and Amend, New York. The description will, however, enable anyone to ascertain if a particular instrument complies with the requirements laid down by the author as essential for accurate work.

The instrument is standardised on water at several temperatures, or with ether or hexane for very fluid liquids, or with sugar solutions for very viscous ones (*cf.* Hoskins, *Phil. Mag.*, 1900, [5], **49**, 274).

The formula for calculating viscosity is—

$$\eta = \frac{\pi g r^4 t p}{8 V l} - \frac{m n \rho V}{8 \pi t l},$$

where g is the acceleration due to gravity, r is the radius of the capillary in cm., t the time in seconds, p the pressure in grm. per sq. cm., V the volume of flow in c.c.,

l the length of the capillary. The second term contains the correction for the loss of kinetic energy, and it should always be but a small fraction of the whole, preferably less than 1 per cent. The number of capillaries is represented by n, while ρ is the density of the liquid under investigation, and m is a constant equal to 1·12. For a given instrument the equation becomes $\eta = Ctp - C'\rho/t$. Since the second term is small compared with the first, the value of $C' = mnV/8\pi l$ may be obtained by approximate measurement with sufficient accuracy. Knowing the values of p and t, as well as the density and viscosity of the liquid used in standardising, the value of C may be readily calculated. Certain minor corrections are necessary, one of them involving the density of the liquid, as does the kinetic energy correction; but as both these corrections are very small in a properly proportioned instrument, the density need only be known approximately, which constitutes a great advantage of the method.

Apparatus for securing widely varying pressures and measuring these with precision is figured in the text. G. C. J.

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REPORTS.

Tests of Permissible Explosives. C. Hall and S. P. Howell. (Bulletin 66, *Bureau of Mines*, Washington, 1913.)—The fifteenth bulletin of this series (ANALYST, 1912, **37**, 290) described the work done at the Pittsburg Testing Station, to which may be largely attributed the steady substitution, since its foundation in 1908, of permissible explosives for others less suitable for use in fiery mines. The present volume, of 313 royal octavo pages, continues the record; it gives an account of new methods and apparatus, with drawings, and detailed results of a large number of tests on permissible explosives and on six foreign explosives, the tests on which were correlated with the Woolwich standards. A pendulum friction machine, for determining sensitiveness to a glancing blow, and a large impact machine, with a dropweight of 200 kgrms., have been installed; the new impact machine gives results more consistent and more nearly representing the sensitiveness of the explosive than the old smaller machine. O. E. M.

Summary of First Report of the Committee on the Nomenclature of Alloys. (Presented at the Annual General Meeting of the Institute of Metals, held in London on March 18, 1914.)—The Committee, consisting of representatives of the Institute of Metals, the Institution of Mechanical Engineers, the Institution of Electrical Engineers, the Institution of Naval Architects, the Institution of Engineers and Shipbuilders in Scotland, the North-East Coast Institution of Engineers and Shipbuilders, and the Society of Chemical Industry (Chairman, W. Rosenhain), present a unanimous report, in which they recommend the adoption, first, of a systematic nomenclature in which alloys are denoted by the names of their constituent metals in English, arranged in increasing order of percentage present in the alloys. This systematic nomenclature is intended chiefly for scientific and other purposes where its precise character more than outweighs its cumbrousness. The Committee next recommends that a system of "practical" nomenclature should be set up, consisting of "practical" names which are to serve as everyday abbreviations of the systematic or scientific names. The definition of current practical names on this basis has so far only been attempted in regard to the two important terms "brass" and "bronze." The definition of "brass" adopted by the Committee is that it is to be used as an abbreviation of the words "zinc-copper" as employed in the systematic nomenclature; when employed alone, it indicates that the alloys are pure zinc-copper; where the presence of other metals is to be indicated, their names are to be prefixed to the term, forming such words as "tin-brass," "aluminium-brass," "manganese-brass," etc. Similarly, the term "bronze" is defined as an abbreviation for "tin-copper" as used in the systematic nomenclature, the definitions in other respects being identical as that for "brass." The Committee is not yet prepared to recommend definitions of further practical terms, but the two terms defined represent the most widely used alloys, and their general adoption as thus defined would do much to remedy the state of confusion which exists at the present time. The Committee therefore appeal to all those interested in the progress of the industries and sciences connected with metals to use their best endeavours to support and carry out the recommendations of the Committee.

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LAW REPORT.

Cattle and Poultry Food: "Artificial Preparation." Worcestershire County Council *v.* Notley Brothers. (King's Bench Division; before Mr. Justice Avory, Mr. Justice Rowlatt, and Mr. Justice Shearman. From the *Times*, May 2, 1914.)—This, a case stated by justices for the county of Worcestershire, raised a point of interest to buyers of cattle and poultry food.

On September 13, 1913, an information was preferred by Richard Griffin (official sampler under the Fertilisers and Feeding Stuffs Act, 1906), duly authorised in that behalf by the Worcestershire County Council (the appellants) under the Act, against the respondents for that they in selling for use as food for cattle or poultry a certain article, to wit, 3½ lb. weight of sharps, to the said Richard Griffin, unlawfully did fail without reasonable excuse to give to the purchaser, on or before, or as soon as possible after the delivery of the said article (such article being an article artificially prepared otherwise than by being mixed, broken, ground, or chopped), the invoice required by the Act stating the respective percentages of oil and albuminoids contained in the said article. On October 21, 1913, the justices, sitting as a Court of summary jurisdiction, heard the information and dismissed it, but agreed to state a case. There was annexed to the summons served on the respondents, as required by section 6 (4) of the said Act, a copy of the certificate given to the prosecutors by the agricultural analyst for the county of Worcester, stating the result of his analysis.

Upon the hearing of the case it was found:

(*a*) That the sharps referred to in the information were purchased on June 21, 1913, by Griffin for use as food for cattle or poultry, and that an invoice was given to him by the respondents which did not state the respective percentages of oil and albuminoids contained in the article.

(*b*) That the information was laid by the authority of the appellants and with the consent of the Board of Agriculture, as required by section 6 (3) of the said Act after a certificate of analysis by the chief analyst had been given.

(*c*) That sharps are an offal of wheat and are that part of the wheat which remains after the flour and bran have been removed.

(*d*) That sharps are made in the following way: Wheat is taken in the whole grain and passed by the miller through his mill. The wheatmeal thus made is then by mechanical means in the mill passed by air currents through a series of sieves whereby it is separated and divided into three substances—namely, flour, sharps, and bran. The chemical composition of each of these three substances (when made) differs both from that of each other and from that of the original wheat. Sharps contain a larger percentage of oil and albuminoids than flour.

(*e*) That the mechanical process does not otherwise effect any chemical change.

(*f*) That the sharps sold by the respondents were a good average sample.

(*g*) That sharps are almost universally of even quality, both in respect of oil and albuminoids.

The justices decided that sharps were not by being separated from flour and bran by the process above mentioned artificially prepared otherwise than by being mixed, broken, ground, or chopped within the meaning of Section 1 (2) of the said Act, and they dismissed the information.

The question for the Court was whether this decision was right.

Mr. Tindal Atkinson, K.C., and Mr. H. S. Cautley appeared for the appellants; Mr. Jenkins, K.C., and the Hon. R. Coventry appeared for the respondents.

The Court delivered a reserved judgment to-day.

JUDGMENT.

Mr. JUSTICE AVORY read the following judgment: The question for decision is whether an article known as "sharps," which was sold for use as food for cattle or poultry, is an article "artificially prepared otherwise than by being mixed, broken, ground, or chopped" within the meaning of Section 1 (2) of the Fertilisers and Feeding-Stuffs Act, 1906. The decision of this Court in Latham *v.* Spillers and Bakers, Limited ([1913], 2 K.B., 355), to which reference is made in the special case, does not, in my opinion, govern the present case. The article sold in that case was composed of various substances, some of which had been artificially prepared, and the contention was that as the different substances had been merely mixed together, the article so produced was not an article artificially prepared otherwise than by being mixed. There are, however, expressions of opinion in the judgment as reported of Mr. Justice Ridley not necessary to the decision, upon which reliance has been placed in the argument for the appellants, the effect of which is that any article in a condition other than as nature made it is artificially prepared within the meaning of this section, if it is prepared otherwise than by being mixed, broken, ground, or chopped. I adhere to what I said in Latham *v.* Spillers and Bakers, Limited (*ubi sup.*): "The section, as a whole, applies to articles artificially prepared. This Act was passed because of a practice of selling for food substances the composition of which was hidden from the purchaser. The object of the Act was to inform the purchaser as to the nature of the substance which he was buying."

"Sharps" is a well-known substance, and as found in the case is that part of wheat which remains after the flour and bran have been removed. The flour, sharps, and bran are produced by a process of grinding and sifting, whereby the grain of wheat is separated into these three component parts, and no chemical change is effected by the process in the component parts. It follows, if the argument of the appellants is right, that both flour and bran, produced by the same process, are "articles artificially prepared otherwise than by being mixed, broken, ground, or chopped." It would follow also, I think, from the argument, that hay which is prepared by a process of cutting and drying would be an article artificially prepared within the meaning of the section, although chopped hay would appear to be just such an article as is contemplated in the exception. I think that some substances, as hay, flour, sharps, and bran, are clearly not within the object of the Act. The question is whether they, or sharps in particular, are within the plain meaning of the words.

In my opinion, although I admit the difficulty of construing the words, the section cannot mean that any article prepared otherwise than by being mixed, broken, ground, or chopped—as for instance peas separated from the pod and dried, or eggs hard boiled—is to be deemed artificially prepared within the meaning of the section. I think that natural substances such as flour, sharps, and bran, the nature of which is well known to the purchaser and which are produced merely by a process of separation without any chemical change, are not artificial preparations within the meaning of the Act, and on this ground I should uphold the decision of the justices in dismissing the information. If the words of the section compelled me to hold that sharps or flour or bran was an artificial preparation, I should also hold that it was not prepared otherwise than by being ground within the meaning of the section. I cannot believe that the Act contemplated that samples of hay and bran should be submitted to the Official Agricultural Analyst and the Chief Government Analyst appointed under the Act for analysis. For these reasons I think the appeal ought to be dismissed.

DISSENTING JUDGMENT.

Mr. JUSTICE ROWLATT differed. He said that the phrase used in Subsection 1 was "subjected to any artificial process." In Subsection 2 that phrase was discarded in favour of the phrase "artificially prepared," and where an article had been artificially prepared the purchaser was entitled to a statement whether it had been prepared from one substance or seed or more. That seemed to him to indicate that what was in view was an article "artificially prepared" from another or others.

in the sense of changing the recognisable form of the constituent or constituents. This would exclude the case of hay and the like, but would include the cases which the subsequent part of the section compelled them to treat as capable of inclusion, such as mixtures and the like, and among them the case most material for the present purpose, namely, that of meal of all sorts. He also thought that the sharps had been artificially prepared "otherwise than by being ground."

Mr. JUSTICE SHEARMAN delivered a judgment agreeing with Mr. Justice Avory that the appeal should be dismissed.

JULY, 1914. Vol. XXXIX., No. 460.

THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

AN ordinary meeting of the Society was held on Wednesday evening, June 3, in the Chemical Society's Rooms, Burlington House. In the absence of the President, the chair was occupied by Mr. L. Archbutt, F.I.C., Past-President.

The Chairman stated it had been decided in Council that an announcement should be made that members and others present at ordinary meetings of the Society should be requested not to communicate discussions to the press.

The minutes of the previous ordinary meeting were read and confirmed.

Certificates of proposal for election to membership in favour of Messrs. W. R. Hardwick, B.Sc., F.I.C., H. F. Hills, and R. H. Kay, were read for the second time; and certificates in favour of Messrs. John Kent Crow, D.Sc. (Lond.), F.I.C., 43, Westcombe Park Road, Blackheath, S.E., Managing Director of Messrs. Wilkinson, Heywood and Clark, Ltd., Varnish and Colour Manufacturers; and Horace Finnemore, B.Sc. (Lond.), F.I.C., Pharmaceutical Laboratory, Guy's Hospital, S.E., Pharmacist and Teacher of Pharmacy at Guy's Hospital, were read for the first time.

The following papers were read: "The Symbolical Representation of Analytical Operations," by L. Gowing-Scopes; "The Properties of Some Chlorhydrocarbons and their Uses in Chemical Analysis.—Part II.," by L. Gowing-Scopes; "The Determination of Iridium in Platinum-Iridium Alloys," by C. O. Bannister; "The Changes in the Character of Fats during the Process of Cooking," by Helen Masters and H. Llwellyn Smith, B.Sc., F.I.C.; "The Insoluble Bromide Value of Oils and its Determination," by Alexander Gemmell, B.Sc., A.I.C.; "On the Chief Cause of the Loss of Sulphuric Anhydride and of Chlorine by Ashing Substances containing these Constituents," by James O'Sullivan, F.I.C.; "Note on the Presence of Sulphates in Flour," by R. A. Cripps, F.I.C., and A. G. Wright; and a "Note on Shrewsbury's Reaction for Paraffin," by Herbert S. Shrewsbury, F.I.C.

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THE DETECTION OF CASTOR SEEDS.

BY G. D. LANDER, D.Sc., F.I.C., AND J. J. GEAKE.

(*Read at the Meeting, May* 6, 1914.)

RICIN, the toxic principle of the castor seed, is devoid of characteristic chemical properties. In its capacity as a toxin, it causes the formation in the blood-serum of an immunised animal of a specific antiricin, which besides being antagonistic to the toxin, gives a precipitin reaction with it.

This latter reaction is not only delicate, but is specific. One c.c. of a 1 : 70,000 extract of Merck's ricin in physiological salt solution (0·85 per cent.) still gives a precipitin reaction (Kranich, *vide infra*), yet this is but a rough guide to the real ricin dilution. Purification of ricin extracts may be pushed to such a degree that the toxin is scarcely ponderable, though retaining its full physiological action (Jacoby, and T. von Oppenheimer, "Toxine und Antitoxine," 1904, 163).

The detection of ricin in extreme dilution is a matter of academic interest, but the biological tests as applied to the recognition of castor-seed adulteration of foods have a real value, and possess an advantage over the microscopic methods in their specificity. The shells of the castor seed do not contain ricin, and it might happen that the ricin of the seeds had been "killed" by heat. The biological tests reveal active ricin, but ignore the "killed" toxin.

A considerable amount of work has recently appeared abroad describing practical methods of testing. Particular reference may be made to Miessner (*Mitt. Kaiser-Wilhelm-Inst.*, 1909, **1**, 3), Mooser (*Landwirtsch. Versuchsstat.*, 1911, **75**, 115), and Kranich (*Zeitsch. Veterinärkunde*, 1912, **24**, 455). Miessner used 10 per cent. salt solution for the extraction of ricin, whilst Mooser and Kranich claim that the employment of glycerol gives better results.

Kranich extracts 2 grms. of the suspected material with 40 c.c. of glycerol at 50° C. (ricin being relatively thermo-stable). The extract is filtered, and to the filtrate is added ten times its volume of a mixture of equal parts of alcohol and ether, the precipitate filtered and washed with alcohol and ether. The air-dried precipitate is taken up in 10 c.c. of 10 per cent. salt solution at 50° C. and filtered through asbestos. One c.c. of the filtrate is placed in a tube and 0·1 c.c. of an antiricin serum (Merck) is caused to form a supernatant layer. Experimenting on ricin mixtures made from pressed seeds and common food-stuffs, Kranich noted the production of turbidity within ten to twenty minutes with proportions of castor seed as low as 0·1 per cent. In each case, after twelve to eighteen hours, a deposit forms at the bottom of the tube, and the size of this deposit is a rough guide to the relative proportion.

Our procedure differs only in detail from that of Miessner. We use 40 c.c. of 0·9 per cent. salt solution and 2 grms. of material, incubate for one hour at 37° C., and usually allow to stand overnight. Miessner's chief difficulty appears to have been in obtaining a clear filtrate. We think that we have satisfactorily overcome this difficulty by centrifuging. Our test experiments were made on mixtures of defatted seeds and linseed meal, which we selected as one of the most difficult substances from which to get a clear extract. By centrifuging for an hour at

3,800 revolutions per minute, the separation was satisfactory, and the liquid almost clear. It is desirable to avoid filtration, for we believe that there is risk of loss, especially with porcelain filters, on account of adsorption.

Since we use 0·9 per cent. salt solution, we cause our extracts to form a layer over the antiricin serum (about 0·1 c.c.). A positive test is indicated by the formation of a zone very like that of albumin in the nitric acid test. Care must be taken not to confuse any haze in the liquid with a genuine precipitation. On standing, the zone extends in magnitude, and after about twelve hours forms a deposit. This deposition ought to be noted as an essential part of the test. Good results may be obtained on an extremely small scale by using capillary tubes. One draws the liquids successively into the tube, taking care to prevent liquid contact. Precipitation at the point of juncture is easily visible. In a similar way one can apply general alkaloidal precipitation tests without using more than $\frac{1}{150}$ c.c. of liquid for each test.

We compared our process with that of Kranich, using a 0·1 per cent. mixture. In our judgment there was little to choose between the two results, but naturally the final liquid obtained by the glycerol method was less coloured. One reason for using 0·1 per cent. salt is that the same extract may be used for the well-known blood agglutination test for ricin. The agglutination is a very striking phenomenon, eminently characteristic of ricin, and less so of crotin, and possibly of other vegetable toxins. Although the corpuscles of different species vary, we believe that all those hitherto examined are sensitive to ricin agglutination. Unfortunately a relatively large proportion of protein interferes seriously with the sensitiveness of the test. It is therefore wise to use a dilute suspension of well-washed corpuscles in physiological salt solution. Rapid agglutination is shown by a mixture of 10 per cent. castor and 90 per cent. linseed, but below that proportion the agglutination is not so marked. The test can be applied on a small scale, for instance in a drop, which can be observed under the microscope.

In the event of doubt as to whether ricin is the actual agglutinating agent, the test can be controlled by means of antiricin. Equal amounts of normal serum and of antiricin serum are added to equal volumes of the suspected extract, and then to each an equal volume of the diluted blood-corpuscle suspension. If ricin is present the agglutination is inhibited in the antiricin ricin preparation.

DISCUSSION.

Mr. C. **Revis** asked if the serum could not be dried. Other dried serums had been used with success, and, although there would necessarily be some loss of power, the dried serum would keep for a longer time.

Dr. **Voelcker** thought, however, that, useful as such a test as this might be for confirmatory purposes, it could never be placed in the same rank with the evidence afforded by the actual presence of, and the physical characteristics possessed by, the castor-seed husk itself. Incidentally it might be mentioned that the bleaching test was due to Dr. Leather, who at the time was one of his (Dr. Voelcker's) assistants; and, although the authors were correct in saying that it might sometimes be misleading, that was only when it was not properly followed up. Bleaching was a most useful method of separating all material which was of a suspicious nature, and which might include castor, and he had seen nothing to weaken his confidence in it as a preliminary method. A good deal of further examination was, however, required, the necessary skill in which could only be obtained by actual experience. He had often been asked what quantity of castor-oil seed might be expected to cause harm, but his answer had been that the presence of *any* quantity whatever might indicate danger. In the case, for instance, of linseed cake, it was very unlikely that the castor-oil seed would be distributed equally; it might possibly be contained entirely in a few cakes only. Consequently, it was of little practical value to say that in a small sample, perhaps a mere handful, there was 1 per cent., or 5 per cent., or 10 per cent., this being no guide to what was present in the portion that might have been actually consumed. He noticed that the authors spoke of having used "defatted" castor-oil seed, and he should like to hear whether they had made any experiments with the so-called "extracted" seed, from which the oil had been removed by a solvent. It was claimed that such extraction removed also the poisonous properties of the seed.

Mr. W. PARTRIDGE asked, in the event of the food of the test animal including any constituent—for example, a protein capable of giving a precipitin reaction, and that protein happened to be also contained in the food under examination—whether a fallacious result would be produced.

Mr. F. W. F. ARNAUD asked if the serum could not be prepared by feeding the animals instead of by injection. If that were so, perhaps the easiest way would be to feed guinea-pigs, which generally appeared to eat food containing as much as 2 per cent. of castor-oil seed without ill effect.

Mr. E. R. BOLTON said that in view of the demand for new kinds of oil seeds, caused by the dearness of all kinds of oil, it seemed possible that in course of time there might come into the market some harmless seed which closely resembled castor in its microscopic structure; and in such an event recourse would have to be had to some such test as that now described.

Mr. REVIS remarked that a method of this kind would be vastly more delicate than the usual chemical or other methods. The specificity of precipitin reactions was extraordinary, and he had no doubt that in this case it would be diagnostic of ricin alone, and consequently of castor-oil seed.

Dr. LANDER said that there was no doubt that, as Mr. Revis had remarked, these reactions were much more delicate than chemical reactions, and, moreover, were diagnostic. It might be mentioned that ricin was not an ordinary chemical poison at all, but was a disease, in the same sense in which snake-bite and diphtheria toxin were diseases. When administered by feeding, its toxicity was reduced at least a hundredfold; indeed, most biological poisons were not absorbed into the alimentary tract at all. He was afraid he could not give a complete answer to the question raised by Mr. Partridge, but as far as the effects of the ordinary constituents of food were known he did not think there was reason to suppose that they would make any difference to the results of this test. The sensitiveness of ricin extract to the blood agglutination test was supposed to be 1 in 600,000, while the sensitiveness to the precipitin test was about 1 in 50,000; and the test could be made roughly quantitative, on a similar principle to that of the estimation of albumin in the albuminometer, by marking on the tube the height reached in a given time by the precipitate from a given percentage of extract in a given quantity of serum. He was in practically complete agreement with the remarks made by Dr. Voelcker. The quantitative estimation of castor seed in cattle food was a matter entirely of academic interest. In cases in which harm had been done the contamination would be gross, and visible and tangible evidence of it would be obtainable even without the aid of the microscope. They had not made any experiments with extracted seeds other than pressed seeds. In the case of the seeds which they had themselves defatted, the extraction had been made with ether which did not dissolve the ricin.

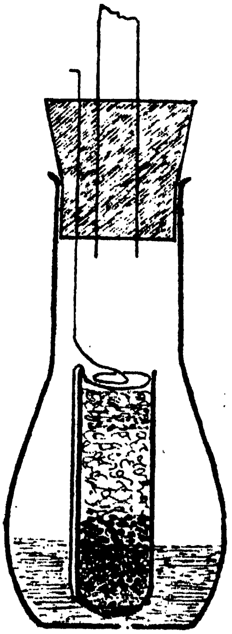
* * * *

A SIMPLE FAT EXTRACTOR.

By G. A. STOKES.

(*Read at the Meeting, May* 6, 1914.)

Most of the fat extractors at present in use appear to have the following defects: (1) The substance to be extracted is placed in a glass tube, so that the outer surface is not exposed immediately to the action of the solvent. (2) The solvent usually exerts its action after it has been cooled by condensation. (3) The extractors are more or less complicated and therefore expensive.



The apparatus I have devised consists of a flask, a piece of wire, and a thimble of fat-free paper. Through the cork connecting the flask with the condenser passes a wire, so bent as to hold a fat-free paper thimble within the flask. Into this thimble the substance to be extracted is placed, and above it a layer of fat-free cotton-wool to prevent any of the substance floating over. The wire is then bent into a spring so as to allow the thimble to be inserted and the cotton-wool pressed down. At the commencement of the extraction the thimble and its contents are forced down by the wire so that they are immersed in the solvent.

After boiling for a short time in the solvent, the hook of the wire on the outside is raised, without disconnecting any part of the apparatus, and the thimble brought out of the liquid, into the neck of the flask. As the space between the neck and thimble is narrow, the boiling vapour as it ascends must act upon the outside of the thimble. The thimble being pervious is constantly being washed inside and out by the hot solvent.

When the extraction is finished, the flask is detached, the solvent evaporated, and the flask and its contents weighed.

The advantages claimed are speed, exactness, simplicity, and cheapness. The thimble can be used a number of times.

Kalusky (ANALYST, 1913, **38**, 48) also uses a thimble, but it cannot be raised or lowered.

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NOTE ON "SHREWSBURY'S REACTION FOR PARAFFIN."

By HERBERT S. SHREWSBURY, F.I.C.

(*Read at the Meeting, June* 3, 1914.)

THIS method (ANALYST, 1909, **34**, 348) has been somewhat misunderstood on the Continent. An abstract of a paper (ANALYST, 1914, **39**, 43) by P. A. Meerburg (*Chem. Weekbl.*, 1913, **10**, 742; through *Chem. Zentralbl.*, 1913, **11**, 1778) states:

"In testing fats for paraffin by the method of Shrewsbury, the saponified mass should be added to the methyl alcohol at once instead of drop by drop. To dissolve the soap the mixture must be warmed and then cooled. If the saponification is carried out in a Jena flask, the glass is attacked by the solution of sodium hydroxide in glycerol, and the solution becomes turbid. A metal vessel is thus better for this purpose. Even then quantities of paraffin below 0·1 per cent. cannot be detected with certainty."

In the original description I stated "the hot mass is dissolved in 50 c.c. of industrial (non-mineralised) methylated spirits, added very gently drop by drop from a pipette."

The spirit should be added to the saponified fat and not *vice versa*, and should be mainly ethyl alcohol and not methyl alcohol. No doubt confusion has arisen through use of the term methylated spirit. Rectified spirits containing 90 per cent. ethyl alcohol could be substituted for the industrial methylated spirits which consist of ethyl alcohol admixed with a small percentage of methyl alcohol.

If the original method is carried out as described, the soap will be dissolved without any further application of heat, unless the air temperature is considerably lower than 18° C.

I have never experienced any difficulty from the action of the alkali on the glass. About three minutes' heating is usually sufficient to effect saponification. The test is not intended to be of a very accurate nature, quantities of paraffin below 0·1 per cent. having little or no significance in the usual examination of a fat.

* * * * *

THE INSOLUBLE BROMIDE VALUE OF OILS AND ITS DETERMINATION.

CARRIED OUT UNDER THE ANALYTICAL INVESTIGATION SCHEME OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

BY ALEX. GEMMELL, B.Sc., A.I.C.

SINCE Hehner and Mitchell (ANALYST, 1898, **23**, 313) published their original paper on this subject, several researches have been carried out, with the result that tables of constants have been published from time to time varying within wide limits. The result has been to render the determination of this figure valueless as a means of detecting adulteration in the classes of drying, semi-drying, and marine animal oils to which these methods apply. The object of this addition to the somewhat extensive literature on the subject has been to investigate the method most likely to give constant results with the easiest manipulation, and, if necessary, to lay down a proposed standard method for its determination.

The methods previously published may be divided into two classes — viz., (1) Those in which the glycerides are themselves directly brominated as recommended by Hehner and Mitchell (*loc. cit.*), Proctor (*J. Soc. Chem. Ind.*, 1906, **25**, 798), and lately Sutcliffe (ANALYST, 1914, 28); and (2) those in which the fatty acids liberated from the glycerides are brominated, as first recommended by Lewkowitsch, without, however, giving details of the method employed, and later by Bull and Johannsen (*Chem. Zeit.*, 1909, 73), and Eibner and Muggenthaler (ANALYST, 1913, **38**, 158).

On the nature of the precipitate obtained, little work has been done except by Proctor (*loc. cit.*), who, however, confined himself to the marine animal oils.

The original method of Hehner and Mitchell was first tried, a sample of refined linseed oil being used. Four determinations were made using 35 c.c. of ether and 5 c.c. of glacial acetic acid, and three using 40 c.c. of ether without acetic acid. The following results were obtained:

Grms. of Oil.	Grms. of Bromide found.	Percentage of Bromide.	Percentage of Br_2 in Bromides.
1·5000	0·5539	36·93	—
2·2850	0·9065	39·66	56·44
2·2525	0·9105	40·41	58·58
2·2790	0·9230	40·50	57·94
2·3370	1·0275	44·74	58·11
2·1750	0·8970	41·34	56·04
2·1520	0·9690	45·03	57·00

The bromine was determined as follows: 0·5 grm. of the bromides was mixed with 2 grms. of finely powdered soda lime, and gently heated over a rose burner in a porcelain crucible for thirty minutes. The sintered mass was extracted with boiling water, filtered, the filtrate cooled and acidified with dilute nitric acid, excess of $\frac{N}{10}$ silver nitrate added, and the excess titrated back with $\frac{N}{10}$ potassium thiocyanate, using ferric alum as indicator.

It is apparent from the above results that using Hehner and Mitchell's method, variation in the results may be due to at least two causes—viz., according to the weight of oil taken, and also according to the proportion of glacial acetic acid used. It is also to be noted that the bromine content of the precipitate varies in apparently no definite manner. In the absence of acetic acid, it was also observed that the precipitate was not in a crystalline condition, and was much more difficult to filter and wash than when acetic acid was originally present, and on drying became brown. Still another point noticed was that sometimes a difficulty arose in the first washing given to the precipitate, owing to the wash liquor (glacial acetic acid) crystallising. Even the highest figure obtained for the percentage of bromine in the bromides varied considerably from that corresponding to the hexabromoglyceride (viz., 62·28 per cent.), of which the precipitate is supposed to consist.

Experiments were next tried using different solvents from those previously recommended.

(*A*) *Petroleum Spirit.*—The oil was dissolved in 30 c.c. of petroleum spirit and 1 c.c. of bromine, added drop by drop. This should precipitate both tetrabromo- and hexabromo-glycerides. The mixture after standing was decanted, and the precipitate washed by decantation with cooled ether containing 5 per cent. of glacial acetic acid, and finally filtered through a Gooch crucible. By this treatment, the tetrabromoglyceride should be removed, and the hexabromoglyceride remain. Great difficulty, however, was experienced in washing the precipitate free from bromine, and filtration was slow. On drying, the precipitate became brown and sticky. The following results were obtained:

Oil taken, 2·3075 grms.	Percentage bromides, 37·70
,, 2·3590 ,, ...	,, 35·93

(*B*) *Alcohol-Ether.*—The oil was dissolved in 15 c.c. of a mixture of equal volumes of ether and rectified spirits, and 1 c.c. bromine dissolved in 15 c.c. of the alcohol-ether mixture, and after cooling added to the solution of the oil. After standing, the brown liquid was decanted, the precipitate was washed with cooled ether, and filtered through a Gooch crucible. The product was difficult to filter and wash free from bromine, and dried brown and sticky.

(*C*) *Chloroform.*—All the bromo derivatives were found to be soluble in this solvent, even on cooling to −5° C. and standing for four hours.

From these series of experiments it is clear that no advantage is to be gained by the use of any of the other solvents tried. Variations in the method of precipitating the bromide with the oil dissolved in ether were next tried.

Series A.—Nos. 1 to 3: Oil in 25 c.c. of ether, cooled, and 1 c.c. of bromine dissolved in 15 c.c. of ether added.

Nos. 4 to 6: Oil in 20 c.c. of ether, and 5 c.c. of glacial acetic acid, cooled, and 1 c.c. of bromine in 15 c.c. of ether added.

No.	Grms. of Oil.	Grms. of Bromide found.	Percentage of Bromides.
1	1·5925	0·8240	51·74
2	1·6530	0·8685	53·14
3	1·6195	0·8675	53·56
4	1·4835	0·8390	56·55
5	1·4630	0·8335	56·97
6	1·4240	0·7595	53·34

The precipitates were washed as follows: First, with 10 c.c. of cooled ether containing 5 per cent. of glacial acetic acid, followed successively by 10 c.c. and 5 c.c. of cooled ether, and 5 c.c. of cooled rectified spirits (twice). The precipitates dried to a good colour, but in quantity showed variations, of in some cases 3 per cent.

Series B.—Six lots of oil in 35 c.c. of ether and 5 c.c. of glacial acetic acid. Bromine was added drop by drop till in excess. Cooled, filtered, and washed as in previous series.

No.	Grms. of Oil.	Grms. of Bromide found.	Percentage of Bromides.
1	1·4045	0·5820	41·43
2	1·3930	0·5430	38·99
3	1·4370	0·5645	39·34
4	1·4225	0·6110	42·94
	1·3930	0·6175	44·33
	1·4680	0·6110	41·62

The variation between the results in this series are as great as in the previous one, while the actual percentage of bromide obtained was much less.

Series C.—Six lots of oil in 20 c.c. of ether. To Nos. 1 to 3, 15 c.c., and to Nos. 4 to 6, 10 c.c. of a mixture containing ether 170 c.c.; bromine, 10 c.c.; glacial acetic acid, 10 c.c. Cooled, filtered, and washed as in previous series.

No.	Grms of Oil.	Grms of Bromide found.	Percentage of Bromides.
1	1·4605	0·7750	53·10
2	1·4610	0·7760	53·11
3	1·4925	0·7900	52·93
4	0·7370	0·3090	41·92
5	0·7610	0·3285	43·03
6	0·7410	0·3100	41·83

The results obtained by this method showed that, using approximately the same weight of oil, greater constancy resulted than in the previous two series, and led to the belief that if the weight of oil under examination was kept constant, variations in the individual determinations might be reduced, and so allow of a reliable method being devised.

It was found on further examination that the mixture of ether, bromine, and glacial acetic acid did not keep for more than a few hours, and the following slight variation was made in the procedure. The oil was dissolved in 18 c.c. of ether and 2 c.c. of glacial acetic acid, cooled, and a cooled mixture of 19 c.c. of ether and 1 c.c. of bromine slowly added. The precipitate in every case was filtered and washed according to the method described in *Series A*.

Series D: Effect of Time on Percentage of Bromides.—Lots of 2 grms. of linseed oil were weighed into Erlenmeyer flasks and treated as above, and the brominated solutions allowed to stand in ice-water for various times.

Time in Hours.	Grms. of Bromide found.	Percentage of Bromides.
1	0·6520	32·60
1	0·8340	41·70
2	0·9390	46·85
2	0·9130	45·65
4	0·9650	48·25
4	0·8730	48·65
6	0·9480	47·90
6	0·9400	47·00
10	0·9580	47·90
10	0·9650	48·25
18	0·9425	47·12

It is apparent from these figures that much greater constancy can be obtained by the use of a definite weight of the oil under examination than was possible if any arbitrary amount was used. It should also be noted that variations in consecutive results of at least 1 per cent. were found unless the brominated solution was allowed to stand for a period of at least four hours.

Series E: Effect of Quantity of Oil Taken on Percentage of Bromides.—Weighed quantities of linseed oil, varying from 0·5 to 2·5 grms. in flasks, treated as in *Series D*, allowed to stand in ice-water for four hours.

Grms. of Oil.	Grms. of Bromide found.	Percentage of Bromides.
0·5	0·1410	28·20
0·5	0·1675	33·50
1·0	0·3670	36·70
1·0	0·3490	34·90
1·5	0·6355	42·37
1·5	0·6650	44·33
2·0	0·9785	48·92
2·0	0·9720	48·60
2·5	1·3170	52·68

A glance at the results of this series at once shows that the variations previously found in determining this value were mainly due to the solubility of the bromine compounds obtained. Naturally, the smaller the amount of oil taken for the determination, the lower the result, and the greater the variation between any two results of experiments carried out side by side. It is to be noted that 2 grms. of oil gave less variation than smaller quantities, while the quantity of precipitate to be freed from bromine was not too great. Larger amounts of oil, although giving higher values, were not so easily washed.

When this point in the investigation was reached, the author thought that, whatever the nature of the precipitate was, the problem of obtaining a constant was solved by this method, and at once set about examining several oils, with the following results :

In each case 2 grms. of oil were treated, and the brominated mixture was allowed to stand for four hours in ice-water.

No. 1.—Sample of Refined Linseed Oil. Iodine value, 173·5.

Average of sixteen determinations, 37·40 per cent. bromides, of which—

1 lay between 34·5 per cent. and 35 per cent.
3 „ „ 35 „ „ 36 „
36 „ „ 37
37 „ „ 38
38 „ „ 39
41 „ „ 41·5

No. 2.—Sample of Raw Linseed Oil. Iodine value, 171·7.

Average of fourteen determinations, 37·54 per cent. bromides, of which-

2 lay between 35 per cent. and 36 per cent.
4 „ „ 36 „ „ 37 „
2 „ „ 37 „ „ 38 „
4 „ „ 38 „ „ 39 „
1 „ „ 39 „ „ 40 „
1 „ „ 40 „ „ 41 „

No. 3.—Sample of Boiled Linseed Oil. Iodine value, 168·7.

Average of twelve determinations, 32·29 bromides, of which—

2 lay between 30 per cent. and 31 per cent.
2 „ „ 31 „ „ 32 „
7 „ „ 32 „ „ 33 „
1 „ „ 35 „ „ 36 „

No. 4.—Sample of Boiled Linseed Oil. Iodine value, 184·9.

Average of ten determinations, 45·25 bromides, of which—

2 lay between 43 per cent. and 44 per cent.
1 „ „ 44 „ „ 45 „
5 „ „ 45 „ „ 46 „
2 „ „ 46 „ „ 47 „

From these results it is apparent that, as in previous cases, absolute constancy cannot be obtained. It is to be noticed, however, that the majority of the results lay within 1 per cent. on either side of the average of the whole series. It would, however, be impracticable to carry out such a number of determinations where a large number of examinations was periodically made. It was also observed that one series of determinations might give results differing in some cases by 2 per cent. or more from those obtained in a previous series where the oil under examination was the same, and the procedure adopted, as far as could be judged, identical in both cases. It appears that the personal factor enters too largely into the determination to allow of great constancy in the results obtained. By constant practice and using identical methods, two investigators might obtain concordant results which would possibly be quite different from each other's as a whole.

The same criticism applies to Sutcliffe's method (*loc. cit.*). When this method appeared, although the author had given up the investigation of this problem from the point of view of the examination of the glycerides, he went back on his results and tried several determinations carried out according to the method described, with, however, no greater success. Sutcliffe himself states also that the factor (0·63 iodine value − 78) may vary with different investigators, and if such is the case, it would be impossible to lay down constants for the guidance of analysts as a means of detecting adulteration. Still another objection is the use of no constant temperature, that of tap-water, as he recommends, being liable to great seasonal variations.

The next method tried was a variation of Proctor's (*loc. cit.*), who recommended bromination of a solution of the oil in carbon tetrachloride and subsequent precipitation by the addition of absolute alcohol. The latter substance, however, is so hygroscopic that differences would be liable to arise. The following method was adopted in its place. Two grm. lots of linseed oil were dissolved in 20 c.c. of chloroform-ether mixtures containing various proportions of the two solvents, 2 c.c. of glacial acetic acid added, and, after cooling, brominated with 20 c.c. of ether containing 1 c.c. of bromine. The following results were obtained:

	Per Cent. Bromides using 20 c.c. Chloroform.	Per Cent. Bromides using 10 c.c. Chloroform and 10 c.c. Ether.	Per Cent. Bromides using 15 c.c. Chloroform and 15 c.c. Ether.	Per Cent. Bromides using 20 c.c. Ether.
	25·65 24·75 24·85	28·20 29·45 28·55	35·90 35·10 35·90	35·20 35·65 36·55
	25·08	28·73	35·63	35·80
Average per cent. bromine in bromides ...	59·23	55·98	54·28	53·62

Here, again, there was apparent uniformity in the results, but this disappeared on carrying out a larger series of experiments. One point, however, was made clear by an examination of the percentages of bromine in the precipitates obtained. The varying percentages obtained from the precipitates using different chloroform-ether mixtures, at once disproved the theory advanced that the low percentage of bromine found may be due to the fact that it consisted of the bromo-derivative of a mixed glyceride. Were this the case a fairly constant percentage of bromine in the precipitate obtained from any one oil, however precipitated, would be expected. These figures point rather to the fact that the precipitate consisted essentially of a mixture of the tetra and hexabromoglycerides varying in proportion with the solvent used. It was also apparent from these results that neither was the tetrabromoglyceride so readily soluble in ether, nor was the hexabromoglyceride so sparingly soluble in this solvent as is usually stated in textbooks.

A further examination of the precipitates obtained by the general method previously mentioned was made to ascertain whether the variation in the percentage of bromides obtained was compensated by a corresponding variation in the percentage of bromine in the precipitate. If this were the case, then the product of the percentage of bromide obtained, and the percentage of bromine in the bromide, should be a constant. This, however, was not found to be the case, the percentage of bromine in the precipitate being usually found to be fairly uniform.

Attention was next directed to the fatty acids liberated from the glycerides as a means of solving the problem. The method suggested by Eibner and Muggenthaler (*loc. cit.*) was tried, but found altogether too cumbersome. Difficulty was also experienced in keeping a constant temperature of −10° C., and it was found that unless very carefully carried out, losses were apt to occur owing first to incomplete washing of the anhydrous potassium sulphate over which the ethereal solution of the fatty acids was dried, and secondly, to oxidation of the acids being apt to occur unless dried in a current of hydrogen, which is not always available. Further, the determination took at least three days to complete.

Four 1 grm. lots of linseed oil were saponified with alcoholic potash, evaporated to dryness and dissolved in water. The acids were liberated with sulphuric acid and extracted with 30 c.c. in all of ether, 2 c.c. of glacial acetic acid was added, the whole cooled in ice-water, bromine added drop by drop till in excess, and the mixture allowed to stand for some time in ice-water. The ether was decanted through weighed papers, 10 c.c. of cooled ether added and decanted, the same operation repeated three times with 5 c.c. of cooled ether, and finally washed on the paper twice with 5 c.c. of cooled ether; dried and weighed. *Percentage Bromides:* 20·90 per cent., 21·90 per cent., 19·70 per cent., 19·40 per cent., containing 62·10 per cent. bromine.

These results led to the belief that with modification this method might give accurate results, while it is evident that the percentage of bromine in the precipitate more nearly approached the theoretical (63·36 per cent.) than the precipitate obtained from the glycerides.

The following method was then tried: 5 grms. of oil were saponified with alcoholic potash, the soap dried, dissolved in 100 c.c. of water, and the fatty acids liberated. The whole was cooled under the tap, and about 30 c.c. of ether added, shaken till clear, and the ethereal layer separated. The aqueous portion was re-extracted with about 20 c.c. of ether. The whole ethereal extract was made up to 100 c.c. Four lots of 20 c.c. were pipetted into flasks, 2 c.c. of glacial acetic acid added to each, the solution cooled in ice-water, brominated, and allowed to stand. The brown liquids were decanted, and the precipitates washed thrice in the flasks with 5 c.c. of cooled ether, and twice after transference to weighed papers with the same quantity, dried, and weighed. *Percentage Bromides :* 27·50 per cent., 28·30 per cent., 28·10 per cent., 28·25 per cent., containing 62·14 per cent. bromine.

This method appeared satisfactory, and was found to give concordant results with consecutive series of experiments. On further investigation, slight variations in the method were made, but on the whole it was found to work more satisfactorily than any other tried, while the whole process could be conveniently carried out in one day.

Effect of Time.—Experiments were conducted to ascertain the minimum time which the precipitates should stand to allow of complete precipitation taking place. The results showed that after two hours no further precipitate appeared on allowing to stand for a further period of two hours. A further secondary precipitate appeared in the case of the vegetable oils after standing for several hours, but on examination was found to consist of very impure hexabromide, and was rejected. The quantity of this second precipitate varied in amount, and might to some extent be due to secondary changes taking place on standing, as it was observed that a large amount of hydrobromic acid seemed to be generated at the same time.

Effect of Concentration.—It was found that the percentage of precipitate obtained varied according to the concentration of the solution of acids brominated—the greater the concentration, the larger the percentage. Twenty c.c. of an ethereal solution containing the acids from 5 grms. of the oil in 100 c.c. was found to be the most convenient however, as it gave a precipitate which, even in the case of oils giving 30 to 40 per cent. of bromides, could be easily washed free from bromine. With greater concentrations the precipitate was not so pure, in every case investigated the percentage of bromine being considerably less than that required by theory. In the case of linseed oils, also, where fairly large precipitates were obtained, although higher values resulted by the use of solutions containing the acids from 2 grms. of oil, the precipitates obtained from such a quantity were invariably too large to be conveniently washed free from bromine, nor were they as pure as those obtained by the use of a solution containing the acids from 1 grm. With oils yielding lower percentages of bromides, the use of greater concentrations of the acids would give results more nearly approaching the actual percentage of hexabromide that ought to be obtained, but if the method is to be reliable, the quantity of oil under examination should be kept constant in every case. That the solubility of the bromides in ether has to be taken into account was shown by the fact that concentration of the filtrates and subsequent cooling brought down a further crop of bromides, which, however, were not so pure as the first crop. It is evident, from the study of the bromine content of the fractions, that in every case where both linolenic and linoleic acids were present in the oil, the precipitate consisted essentially of a mixture of hexabromide and tetrabromide, varying according to the proportion of each acid present.

To prove this point, samples of pure linoleic and linolenic acids were obtained, and a series of experiments conducted, various mixtures of the two ethereal solutions of the acids being used, with the following results:

Linoleic Acid.

Grm. of Acid.	C.c. of Glacial Acetic Acid.	Total Volume of Solution.	Grm. of Bromide.
0·2136	nil	10 c.c.	0·036
0·2136	nil	20 „	0·028
0·2136	2·0	10 „	0·027
0·2136	2·0	20 „	0·023

Linolenic Acid.

Grm. of Acid.	C.c. of Glacial Acetic Acid.	Total Volume of Solution.	Grm. of Bromide.	Percentage of Actual Weight required by Theory.
0·2518	nil	10 c.c.	0·098	14·40
0·2518	2·0	10 „	0·065	9·54
0·2518	2·0	20 „	0·058	8·50
0·5036	nil	10 „	0·256	18·80
0·5036	2·0	20 „	0·198	14·59

Linoleic and Linolenic Acid.

Grm. of Linoleic Acid.	Grm. of Linolenic Acid.	C.c. Glacial Acetic Acid.	Total Value of Solution.	Grm. of Bromides.	Per Cent. of Actual Weight required by Theory.
0·2136	0·2518	nil	10 c.c.	0·169	24·82
0·2136	0·2518	2·0	10 „	0·146	21·44
0·2136	0·5036	nil	10 „	0·340	24·96
0·2136	0·5036	2·0	10 „	0·293	21·51
0·2136	0·2518	nil	20 „	0·155	22·76
0·2136	0·2518	2·0	20 „	0·131	19·23
0·2136	0·5036	nil	20 „	0·326	23·93
0·2136	0·5036	2·0	20 „	0·283	20·77

A study of these results shows, first, that even pure linoleic acid gave a precipitate on bromination, even in a 5 per cent. solution containing 10 per cent. of glacial acetic acid; second, that standard conditions as regards concentration of the solution of acids to be brominated and volume of wash liquid to be used, must be laid down, since it is obvious from the results obtained that the chief substance to be dealt with is a soluble one, and results will vary according to the method adopted. Not only is it apparent that different results are obtained by the use of varying concentrations, but also according to whether glacial acetic acid is or is not present. Using solutions containing 10 per cent. by volume of acetic acid, the results were about 16 per cent. lower than those obtained without any acetic acid. The product obtained, however, more nearly approached pure linolenic hexabromide. For example, one precipitate weighing 0·326 grm. contained 60·8 per cent. of bromine without the use of acetic acid, while a similar mixture with the addition of 10 per cent. of acetic acid gave a precipitate which weighed 0·283 grm. and contained 62·4 per cent. of bromine. 60·8 per cent. of bromine corresponds to 25·33 per cent. of linoleic tetrabromide, and 74·77 per cent. of linolenic hexabromide. While 62·4 per cent. of bromine corresponds to 9·31 per cent. of linoleic tetrabromide, and 90·69 per cent. of linolenic hexabromide, so that the loss in weight was compensated for by the greater purity of the precipitate. In all cases, however, where both linoleic and linolenic acids are present, the precipitate consists essentially of a mixture of the bromo derivatives of both these acids.

Effect of Purity of Ether on Amount of Precipitate obtained.—A series of experiments was made, using ordinary commercial ether compared with anhydrous purified methylated ether. It was found that lower results in every case were obtained with commercial ether, and on investigation this was found to be due to the presence of alcohol. The presence of water did not appear to affect the results to any extent. The ether was therefore purified before use by distillation over caustic soda, and then dried as far as possible over anhydrous copper sulphate or potassium carbonate.

It was also found in some cases, where the precipitate was large, that washing with five successive quantities of cooled ether was insufficient to dissolve out all the bromine. The following procedure, however, got over this difficulty: After standing for two hours, the brown liquid was decanted from the precipitate as far as possible, and the residue transferred to a centrifuge tube, centrifuged, and the liquid poured off. Five c.c. of cooled ether were added to the flask, the sides of the flask cleaned with the aid of a rubber-tipped rod, and the liquid transferred to the centrifuge tube. The precipitate and the wash liquid were thoroughly mixed up with a pointed glass rod, centrifuged, and the liquid poured off. This was repeated, then 5 c.c. of cooled ether added to the tube, the whole shaken up and transferred to a weighed paper, the tube washed out with 5 c.c. of cooled ether, and the precipitate finally washed on the paper with 5 c.c. of ether. In this way precipitates were obtained quite free from bromine.

Various oils were examined and the results as given in table on p. 307 obtained.

No exact relationship has been found to exist between the iodine value and the percentage of bromides obtained.

Determination of the Insoluble Bromides in Marine Animal Oils.—It was found on trial that the marine animal oils also gave satisfactory results using the same method as that adopted for the vegetable oils. The precipitates obtained, however, were more finely divided, and it was found advisable to filter through Swedish filter-paper.

	Iodine Value.	Bromides per Cent.
Raw Linseed Oils—		
Refined (origin unknown)	182·5	34·00
Raw "	182·0	33·65
Matured Baltic	197·5	37·65
Baltic (Sample A)	196·0	36·10
" (" B)	184·7	32·60
Plate (" A)	176·5	33·30
" (" B)	175·6	32·60
Boiled Linseed Oils—		
Baltic	170·5	25·95
Plate	165·5	27·75
Sample A	184·9	33·90
" B	180·2	30·20
" C	176·3	27·10
D	172·5	26·65
Other Vegetable Oils—		
Soya bean ...	141·8	4·10
Rape (Sample A)	108·0	2·35
Rape ? (Sample B)	122·8	5·80
Walnut	148·2	3·00
Tung	164·2	nil.

The solubility factor, as far as could be ascertained, was not so great as in the case of vegetable oils, a precipitate appearing whenever bromine was added. This fact could be made use of as a quick and reliable qualitative test for fish oils in vegetable oils. As regards the percentage of bromine in the bromides, this varied from 67 to 68·5 per cent., compared with 70·96 per cent. theoretically required for the compound $C_{17}H_{26}O_2Br_8$.

The following marine animal oils were examined:

	Iodine Value.	Bromides per Cent.
Cod-liver oil...	172·2	35·20
Whale oil	149·3	21·70
Brown whale oil	139·6	25·80
Menhaden oil	170·8	51·70
Shark-liver oil	119·1	17·70
Seal oil?	166·9	29·12
Sperm oil	81·2	1·70

Here, again, there appears to be no exact relationship connecting the iodine value and the percentage of bromides.

The following is a brief summary of the conclusions arrived at by the study of this problem:

1. It has been shown that the solubility factor occupies a very prominent place in the determination of this value, and on this account a standard method of working should be laid down.

2. The direct examination of the glycerides does not yield such good results as working with the fatty acids themselves. This may be due in part to secondary reactions taking place when the glycerides are used.

3. That the precipitates obtained do not in the case of the vegetable oils consist of the pure hexabromide, or in the case of the marine animal oils, of the pure octabromide, but depend largely on the proportions of the various unsaturated acids present in the oil.

4. That the examination of the fatty acids, according to the method recommended in this paper, gives precipitates more closely approximating to the pure hexabromide or octabromide than is the case when the glycerides are directly brominated.

Now that the actual method of determining the insoluble bromide value has been investigated, the author intends to proceed further, with an examination of mixtures of various oils, with a view to the detection of adulteration by the use of this method, the results of which will be published in due course.

In conclusion, the author desires to acknowledge the aid of a grant from the Society, also to thank Messrs. Craig and Rose, Ltd., of Edinburgh, and Messrs. A. L. Seaton and Co., of Hull, for various samples of oils received.

ERRATUM.—Page 250, line 17, "hydrofluoric acid" should read "sodium chloride."

* * * * *

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Composition of the Radicles of Cacao Beans. E. P. Haussler. (*Arch. Pharm.*, 1914, **252**, 82-89.)—The radicles (*radiculæ*) separated from the cotyledons of cacao beans have the following composition, the figures being calculated into percentages of the dry substance: Total ash, 6·53; fat, 7·08; total nitrogen, 5·16; crude fibre, 5·26; acidity (as tartaric acid), 4·47; dextrose, 0·44; sucrose, 2·13; theobromine, 1·88; caffein, 0·21. The water soluble constituents amount to 23·4 per cent., whilst the soluble ash is 6·26 per cent. and has an alkalinity corresponding with 1·72 per cent. of potassium carbonate. The fat has an iodine value of 48·1 and an acid value of 19·2.

W. P. S.

Estimation of "Cellulose" in Flour as a Measure of the Degree of Bolting. L. Lindet. (*Bull. Soc. Chim.*, 1914, **15**, 384-387.)—According to the author, the estimation of cellulose by extraction with boiling acid and alkali may be accelerated by employing the reagents at a higher concentration than usual. Ten grms. of the meal are freed from fat by extraction; the meal is then treated with 400 c.c. of boiling hydrochloric acid (sp. gr. 1·025) for half an hour. To the liquid are added 50 c.c. of a 10 per cent. solution of hydrated aluminium sulphate, and the whole is made alkaline with ammonia. The cellulose residue thus suspended in the gelatinous alumina is filtered off and washed. The precipitate is washed off the filter into a flask and treated with caustic soda, equivalent to 10 grms. per 100 c.c. of liquid. The solution is heated at 100° C. for one hour, and the digestion of the meal thus completed. The liquid is cooled, neutralised with hydrochloric acid, and the alumina reprecipitated by ammonia. The precipitate is collected on a tared filter, washed, and then treated with dilute hydrochloric acid, which dissolves the alumina, leaving the cellulose; this is washed, dried, weighed, and the weight corrected for ash. The author has employed this method as a means of ascertaining the degree of bolting of flours, and gives in a table the percentages of cellulose corresponding to the various grades. Any of the principal hull constituents might serve a similar purpose as a chemical measure of grading, but it is found that the "cellulose" shows the widest divergence between the values for the different grades. J. F. B.

Colorimetric Estimation of Creatine. E. Baur and G. Trümpler. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1914, **27**, 697-713.)—The authors have investigated the colorimetric method for the estimation of creatine, proposed originally by Jaffé, paying particular attention to the influence of time, temperature, and acid concentration on the inversion of creatine to creatinine, and modify the method previously described by Baur and Barschall (ANALYST, 1907, **32**, 48) by heating 10 grms. of meat extract with 100 c.c. of $\frac{N}{1}$ hydrochloric acid for four hours at 97° C. The actual estimation of creatine in meat extracts is carried out as follows: Ten grms. of the extract are dissolved in water to give 100 c.c. of solution; 5 c.c. of this solution are then treated with 15 c.c. of saturated picric acid solution and 5 c.c. of 10 per cent. sodium hydroxide solution, and after seven minutes the mixture is diluted to 500 c.c., and the coloration compared with that exhibited by a definite depth of $\frac{N}{2}$ potassium bichromate solution (see ANALYST, 1907, **32**, 196). This result gives the quantity of pre-formed creatinine. A second portion of 10 grms. of the extract is then heated with 100 c.c. of $\frac{N}{1}$ hydrochloric acid for four hours at 97° C.; after cooling, 5 c.c. of the solution are neutralised, treated with picric acid and sodium hydroxide, diluted to 500 c.c., and the coloration compared as described. The quantity of creatinine *plus* creatine is thus obtained. The comparisons should be made while the solutions are at a temperature of 17° C. Liebig's meat extract was found to contain from 0·76 to 3·72 per cent. of creatinine and from 2·09 to 5·58 per cent. of creatine. A meat extract prepared by the authors (1 kilo of flesh yielded 35 grms. of extract) contained 8·9 per cent. of creatinine *plus* creatine. W. P. S.

Estimation of Cholesterol in Fats. M. Klostermann and H. Opitz. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1914, **27**, 713-722.)—Practically all solid animal fats are free from cholesteryl esters, the cholesterol contained in the fats being present in the free state; consequently, previous saponification of the fat is unnecessary when it is desired to separate the cholesterol by precipitation with digitonin (ANALYST, 1910, **35**, 256; 1913, **38**, 458; 1914, 84). In cod-liver oil however, about one-half of the total cholesterol is present in the form of esters. In the case of vegetable oils, the greater part of the phytosterol present occurs as esters, and in the detection of vegetable oils in animal fats, by precipitating the phytosterol and cholesterol with digitonin, and identifying the former by the melting-point of its acetate, it is essential to saponify the fat in order to obtain the phytosterol in the digitonin precipitate.

W. P. S.

Unsaponifiable Constituents of Mowrah Fat and the Detection of this Fat in Animal and Vegetable Fats. P. Berg and J. Angerhausen. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1914, **27**, 723-731.)—Mowrah fat yields about 2 per cent. of unsaponifiable substances which, in chloroform solution, have a specific rotation of $[\alpha]_D + 27{\cdot}1^\circ$. When dissolved in ether and then treated with a large volume of absolute alcohol, a small quantity of an inactive substance is precipitated, and the rotation of the soluble portion is increased to $[\alpha]_D = +35^\circ$. The unsaponifiable substances do not consist of phytosterol, and are not precipitated by digitonin (*cf.* ANALYST, 1914, 84). Since lard yields unsaponifiable substances having $[\alpha]_D = -19{\cdot}6^\circ$, the detection of mowrah fat in lard may be ascertained from the optical properties of the separated unsaponifiable substances present. This identification is rendered more simple, both in the case of lard and other animal fats and vegetable fats, from the fact that the unsaponifiable substances of these fats consist largely of phytosterol and cholesterol which are precipitated by digitonin, whilst the remaining substances are optically inactive. For the actual detection, 100 grms. of the lard or other fat are saponified, and the unsaponifiable substances, separated in the usual way, are dissolved in ether, and the solution is poured into 100 c.c. of absolute alcohol; if the mixture is turbid, the presence of mowrah fat is already indicated. The solution is then filtered, the filtrate is partially evaporated to remove the ether, and digitonin is added. The digitonin precipitate is separated, the solution is evaporated, and the residue is extracted with ether; the excess of digitonin remains insoluble. The ethereal solution is then evaporated, and the rotation of the residue is determined in alcohol solution. Pure lard yields but a trace of inactive residue, whilst lard containing 10 per cent. of mowrah fat yields a residue having $[\alpha] = +34{\cdot}9^\circ$.

W. P. S.

Estimation of the Unsaponifiable Constituents of Oils and Fats and of Lanolin by Means of the Digitonin Method. H. Salomon. (*Ber. Deutsch. Phar. Ges.*, 1914, **24**, 189-193; through *Chem. Zentralbl.*, 1914, I., 1854.)—The following percentages of unsaponifiable substances were separated from various fats and oils, the figures in parenthesis denoting the amount of solid substances calculated on the quantity of material itself: Lard, 0·27 (64·4); almond oil, 0·26 (72·2); earthnut oil, 0·46 (84·7); cacao fat, 0·36 (58·3); cod-liver oil, 0·71 (76·9); linseed oil, 1·21 (32·4); olive oil, 0·60 (31·54); castor oil, 0·33 (56·5); sesame oil, 1·37 (99·8); peach kernel oil, 0·35 (60·0). The solid portion of the unsaponifiable substances obtained from sesame oil consisted of 36·8 per cent. of sesamin, and 63·2 per cent. of phytosterol; the sesamin is not precipitated by digitonin. The fluid portion consisted of sesamol, the substance which yields the coloration in Baudouin's reaction.

Wool-fat contains 51·6 per cent. of unsaponifiable substances, 57 per cent. of which is not precipitated by digitonin. Lanolin (a mixture of wool-fat, water, and hydrocarbons) yields 48 per cent. of unsaponifiable substances, 70 per cent. of which are fluid. A lanolin soap examined gave 5·16 per cent. of unsaponifiable substances, of which 54 per cent. was not precipitated by digitonin; the soap therefore contained 10 per cent. of wool-fat. The phytosteryl acetate separated from the vegetable fats and oils mentioned above melted between 124° and 129° C., whilst the cholesteryl acetate separated from lard, cod-liver oil, and wool-fat melted at 113° to 114° C.; in the case of peach kernel oil the acetate melted at 119° C. W. P. S.

Action of Halogens on Oleic Acid and Estimation of the Iodine Value of Fats. W. Mergen and A. Winogradoff. (*Zeitsch. angew. Chem.*, 1914, **27**, 241-244.)—Experiments described in detail have shown that of the various solutions used for estimating the iodine values of fats Waller's is the most and Hübl's the least stable. Chlorine acts more energetically than bromine upon oleic acid. If a mixture of chlorine and iodine be used in equivalent quantities, more chlorine than iodine is absorbed, whilst there is also more or less substitution in the case of the chlorine. This substitution is checked by the presence of hydrogen ions. It is not advisable, however, to add a large excess of concentrated hydrochloric acid, as is done in Waller's solution, since above a certain degree of concentration the oleic acid combines with the hydrochloric acid. In estimating the iodine value of an unknown compound it is recommended that Wijs' method should be employed, and that after the titration with thiosulphate, the product of the reaction should be extracted with cold water, and the aqueous extract titrated with $\frac{N}{10}$ alkali to find the amount of halogen acid. For example, a sample of purest oleic acid was treated for thirty minutes with a Wijs' solution containing 13 grms. per litre of iodine, and an equivalent quantity of chlorine. The iodine value found was 99·95 (theory 89·95), whilst the acid in the aqueous solution corresponded to 4·62 per cent. of substituted iodine. In a parallel estimation with the same oleic acid, but with a Wijs' solution containing an excess of 2 per cent. of iodine over the chlorine, the iodine value found was 90·95, whilst the acid in the aqueous extract corresponded to 0·53 per cent. of substituted iodine. When there was an excess of 10 per cent. of chlorine in the Wijs' solution the iodine value of the oleic acid was 105·40, whilst the acid in the aqueous extract corresponded to 8·31 per cent. of iodine. It was proved that halogen acids were only formed by substitution. C. A. M.

Estimation of Fatty Acids in Soaps. A. A. Besson. (*Chem. Zeit.*, 1914, **38**, 645-647, 686-687.)—It is shown that ether gives more accurate results than petroleum spirit in the extraction of the fatty acids in soap, since it dissolves lactones and hydroxy acids which are insoluble in the latter. The difference in the results obtained with the two solvents may in certain cases amount to several per cent. The sapometer (ANALYST, 1912, **38**, 479) is preferable to a separating funnel for the extraction, and in the case of filled soaps the fatty acids should be first separated in a beaker, then dissolved in ether, and the solution filtered through cotton-wool into the sapometer. It is unnecessary to convert the fatty acids into salts before evaporating the solvent, even in the presence of volatile fatty acids. An allowance may be made for any loss that occurs in drying, by taking two aliquot portions of the ethereal solution. One of these is evaporated on the closed water-bath for forty-five minutes, with occasional blowing, and the residue of fatty acids weighed, then dissolved in alcohol and titrated with standard alkali solution. In a second aliquot portion of the ethereal solution the fatty acids are directly titrated, and from the difference between the results the loss of volatile acids (if any) may be calculated.

C. A. M.

Lathering Capacity of Soap. C. Stiepel. (*Seifensieder-Zeit.*, 1914, **41**, 347; through *Chem. Zentralbl.*, 1914, I., 1709.)—The lathering capacity of soap is estimated in a solution containing 0·6 grm. of fatty acid in 100 c.c. of water. The solution is shaken at 17° to 20° C. (or at 50° to 55° C.), in a 2-litre flask, with a long graduated neck on which is a bulb holding 50 c.c. After being shaken for thirty seconds at the definite temperatures, the flask is inverted and allowed to stand for, say, three minutes, and the percentage of froth read directly upon the scale.

C. A. M.

Analysis of Saffron. R. Krzizan. (*Zeitsch. öffentl. Chem.*, 1914, **20**, 109-114, 121-124; through *Chem. Zentralbl.*, 1914, I., 1855-1856.)—Low results are obtained in the estimation of invert sugar in saffron when sodium sulphate is used to remove the excess of basic lead acetate employed for clarification. From 3 to 4 per cent. more sugar is found when the excess of lead is precipitated with sodium phosphate. Certain genuine samples of saffron yielded 21·75 per cent. of invert sugar, and, after inversion, 23·53 per cent., calculated on the dry substance. The accepted maximum of 20 per cent. of invert sugar therefore appears to be somewhat too low. Genuine saffron frequently contains up to 1 per cent. of sand, and usually yields not more than 6 per cent. of total ash. About 70 per cent. of the ash is soluble in water, and the alkalinity of the soluble ash corresponds with 6·9 c.c. of $\frac{N}{1}$ acid per grm. of total ash.

W. P. S.

Estimation of Starch in Plant Material. W. A. Davis and A. J. Daish. (*J. Agric. Sci.*, 1914, **6**, 152-168.)—The Sachsse method of estimating starch, which is official in the United States (*Bureau Chem. Bull.*, No. 107, and Allen's "Com. Org. Analysis," 4th edition, vol. i., p. 420), is inaccurate when used for foliage leaves, seeds, grain, etc., because such tissues contain pentosans and other substances yielding reducing sugars on hydrolysis which count as dextrose, and also because of the actual destruction of dextrose which occurs during the acid treatment.

O'Sullivan's method (*J. Chem. Soc.*, 1884, **45**, 1), though approximately accurate with purified starch, gives results from 15 to 20 per cent. too low when applied to plant material in general, owing to the loss of dextrin, which is carried down when basic lead acetate is employed to separate tannins, proteins, etc. The authors find that starch in plant material may be satisfactorily estimated by the use of "taka-diastase." This diastase converts gelatinised starch into a mixture consisting entirely of maltose and dextrose, when the time of conversion exceeds six hours at 38° C. A certain amount of dextrin is formed at first, but this all disappears within six hours, the proportion of dextrose steadily increasing at the expense of the maltose as time proceeds, until after fifty hours there is 80 per cent. of dextrose present. Beyond forty-eight hours' digestion slightly lower results are obtained. Since no dextrin is present, lead acetate may be safely used to precipitate proteins, etc. The working details are as follows: About 10 grms. of the material, dried at 100° C., are first carefully dried to constant weight at 100° or 110° C. *in vacuo* over phosphorus pentoxide for twenty-four hours or more. To estimate the starch, the dry material (freed from sugars by extraction with alcohol, and, if necessary, previously extracted with water to remove gums, amylans, etc.), is gelatinised with 200 c.c. of water in a boiling-water bath for half an hour. The solution is cooled to 38° C., and 0·1 grm. of taka-diastase added, together with 2 c.c. of toluene, the whole being then left for twenty-four hours at 38° C., after which it is boiled, and the clear solution above the leaf material is filtered into a 500 c.c. flask. The residue is washed several times by decantation, and the washings passed through the filter-paper until the volume in the flask measures about 475 c.c. From 5 to 25 c.c. of basic lead acetate are then added as required, any large excess of lead being avoided. The solution is then made up to 500 c.c. and filtered; 100 c.c. of the filtrate are placed in a 110 c.c. flask, the excess of lead precipitated by solid sodium carbonate, and the volume adjusted to 110 c.c. Fifty c.c. of the filtrate from the lead carbonate are used for the cupric reduction with Fehling's solution, and another portion read in a 400 mm. tube. The weight of copper oxide corresponding to 1 grm. of maltose and dextrose in Brown, Morris and Millar's tables (*J. Chem. Soc.*, 1897, 275) is then used in conjunction with the optical activity of the solution to obtain the weights of maltose and dextrose in 100 c.c. of the solution. The amounts of starch corresponding to the weights of maltose and dextrose so found are then added together and referred to the dry weight of the original material (dextrose × 0·9 and maltose ÷ 1·0555 equal starch). If the dried ground leaf is bottled before analysis, it is necessary to turn out the whole of the material and thoroughly mix it before analysis; otherwise the proportion of starch present in the material is found to increase considerably towards the bottom of the bottle. One of the principal difficulties in estimating starch in plant material is the imperfect separation of all the gummy substances, etc., even by basic lead acetate, and the resulting disturbing effect on the rotation of the sugars formed from starch. It is generally possible completely to remove such substances by prolonged extraction with water prior to the starch conversion, which is then followed by the addition of basic lead acetate in the usual way. In cases where such treatment fails, a "blank" determination of the material with the taka-diastase omitted should be made. A large amount of supplementary material is included, for which the original paper should be consulted. H. F. E. H.

Variety of Starches. C. Tanret. (*Comptes rend.*, 1914, **158**, 1353-1356.)—The author's experiments confirm the view of Maquernne and Roux that starch is a mixture of amylopectin and amylose, and show, moreover, that these components exist in varying proportions in different varieties of starch. The method of separating the amylopectin from the amylose is based on the fact that the latter is soluble when boiled with water for a few seconds, whilst the amylopectin is scarcely affected. 0·2 grm. of starch, calculated on dry weight, is treated with 200 c.c. of water, which is then heated to the boiling-point. The extraction of the amylose in this way is performed three times, and after each treatment the liquid is allowed to remain several days, until the insoluble matter has settled out. If the solution remains opalescent, the settling should be continued until a portion of the liquid, when heated with an equal volume of water, is found to be completely clear. The solution is drawn off by a pipette. After the last extraction the liquid should show practically no coloration with iodine. The residue of amylopectin is collected, dehydrated by alcohol, dried first over sulphuric acid, then at 110° C., and weighed. The proportions of amylopectin vary from 67 per cent. for chestnut starch to 79·5 per cent. for banana starch. Potato starch, with 73 per cent. of amylopectin, cannot be analysed in the above manner, because the amylopectin is soluble to some extent; in this case a portion of the clear liquid from the first extraction was evaporated to dryness and the amount of amylose estimated. In any starch solution containing dissolved amylopectin, including solutions of "soluble starch," the amylopectin may be recovered by taking advantage of the property which cellulose possesses of withdrawing the amylose from solution. The liquid at a dilution of 2 per 1,000 is soaked up by well-purified cotton-wool, and after one hour the cotton is squeezed. The expressed liquid then contains only amylopectin, which gives a violet to red coloration with iodine, discharged by an excess of water, whereas the cotton, containing the amylose, is coloured pure blue. This amylose cannot be removed from the cellulose by cold water; boiling water removes it partially, and a 5 per cent. solution of sodium hydroxide extracts practically all. J. F. B.

Bromine Thermal Value of Turpentine Oil. A. Heiduschka. (*Chem. Zeit.*, 1914, **38**, 441.)—The bromine thermal method devised by Hehner and Mitchell (ANALYST, 1895, **20**, 146) has been adapted by the author to the examination of oil of turpentine: Twenty c.c. of a chloroform solution containing 0·4 grm. of the sample are treated in a vacuum tube with 1 c.c. of bromine at about 17·5° C., and the rise of temperature determined. The following results were thus obtained with genuine turpentine oils and substances used for their adulteration: Dextro-rotatory turpentine oil, 0·8° C.; "French" oil of turpentine (seven samples), 23·0° to 23·8° C.; German oil (two samples), 16·5° and 17·7° C.; rectified oil of turpentine, 23·1° C.; rosin oil, 13·6° C.; ligroine, −0·85° C.; and petroleum oil, 2·3° C. The method thus appears suitable as a test for turpentine oil, and, in particular, for distinguishing between dextro-rotatory and lævo-rotatory oils. C. A. M.

Reactions of Vanillin. E. P. Haussler. (*Zeitsch. anal. Chem.*, 1914, **53**, 363-371.)—The colorations which are obtained when vanillin is treated with various organic substances in the presence of hydrochloric acid or sulphuric acid are recorded. The tests were carried out by mixing a small quantity of an alcoholic solution of vanillin with a trace of the organic substance, evaporating the mixture, and warming the residue after the addition of a drop of dilute hydrochloric acid or dilute sulphuric acid. The substances employed in the investigation included amines, acid amides, imines, hydrazines, phenols, acids, aldehydes, ketones, proteins, etc. Generally the different classes of substances yielded typical colorations; the amines, acid amides, and imines gave yellow colorations, whilst the phenols gave red or violet colorations. The reactions with *p*-phenylenediamine, phloroglucinol, and resorcinol are very sensitive and capable of detecting 0·005 mgrm. of vanillin. A remarkable reaction is obtained in the case of citric acid; on treating the residue (obtained by evaporating an alcoholic solution of vanillin with the addition of citric acid) with dilute sulphuric acid and warming the mixture, a bright violet coloration is produced which turns green when diluted with water, and red on the addition of ammonia (see also Analyst, 1905, **30**, 247, 340). W. P. S.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Modification of Teichmann's Test for Blood. W. D. Sutherland and G. C. Mitra. (*Biochem. J.*, 1914, **8**, 128-131.)—If the stain be on a hard surface, a fragment is placed on a microscope slide and treated with a drop of a 10 per cent. potassium cyanide solution and a drop or two of an ammonium sulphide solution, covered and examined with a ½-inch objective. If the stain be on a soft fabric, a minute portion is removed, plunged into boiling water for a couple of seconds, and then treated as above. If blood be present, a spot of colour will be seen, the tint of which varies from the faintest pink to cherry red. This spot is brought into focus, the eyepiece of the microscope removed, and the long arm of a Browning's hand spectroscope, modified by Zeiss, inserted into the tube. A piece of paper is held under the short arm of the spectroscope to reflect light for the reading of the wavelength scale, and the absorption bands of cyanhæmochromogen are identified. In rare cases only one band may be seen, but in all cases the method succeeds; for even stains that have been more than half washed out give the characteristic spectrum when so treated. An old stain may require to be soaked in the cyanide solution for about a minute before the addition of ammonium sulphide. The authors have nearly always succeeded in finding enough erythrocytes in a material—even earth—in which the presence of blood has been proved spectroscopically to determine whether the blood was mammalian or not. For the determination of erythrocytes the fragment of stained material is soaked in Vibert's fluid (2 grms. sodium chloride + 0·5 grm. mercuric chloride + 100 c.c. water) for half an hour, and examined with an oil-immersion lens. E. W.

Estimation of Amino-Acids in Blood. L. Lematte. (*Comptes rend.*, 1914, **158**, 1379-1381.)—The new method is based on the fact that phosphotungstic acid, in presence of sulphuric acid, precipitates all the proteins and ammonium compounds, but does not affect the amino-acids. The excess of phosphotungstic acid is neutralised and precipitated by calcium chloride. The excess of calcium is precipitated by potassium oxalate, and the amino-acids remaining in solution are estimated by the formaldehyde titration method of Ronchèse. The following solutions are required: A 30 per cent. solution of phosphotungstic acid, 25 per cent. calcium chloride, 30 per cent. potassium oxalate, and standard $\frac{N}{100}$ sodium hydroxide. A quantity of blood, as large as can be obtained, is placed in a graduated, stoppered tube of 50 c.c. Let the volume of blood be *A* c.c. An equal volume of the phosphotungstic acid and $\frac{A}{25}$ c.c. of sulphuric acid are added, with water to make up 50 c.c. The tube is shaken, a granular precipitate is produced, allowed to settle for fifteen minutes, and filtered off. Ten drops of a strong solution of phenolphthaleïn are added, and the liquid is made alkaline by the addition of caustic soda. The phosphotungstate is precipitated by adding $\frac{A}{2\cdot25}$ c.c. of the calcium chloride solution, the precipitate allowed to stand for half an hour, and $\frac{3A}{5}$ c.c. of the oxalate solution are added without filtering. The whole is allowed to settle for an hour, and the volume of the mixture observed; it is then filtered. Two portions of 10 c.c. each are placed in two tubes and neutralised to a standard pale pink shade. A solution of formaldehyde ("formol," 1 in 2) is prepared such that 2 c.c. have no effect upon the neutral tint when added to 10 c.c. of neutralised distilled water. One c.c. of this formaldehyde is added to one of the tubes, and the contents are titrated with $\frac{N}{100}$ sodium hydroxide until the tint is restored. Ronchèse's correction (0·1 c.c. per 3 c.c. of soda used) is added to the result of the titration, *V*. The amount of amino-acid nitrogen is then calculated by the expression: $\frac{V}{3} \times 0\cdot1 \times 0\cdot000014$. J. F. B.

Detection and Estimation of Monamino-Acids. R. Engeland. (*Zeitsch. Biol.*, 1914, **63**, 470-476; through *Chem. Zentralbl.*, 1914, I., 1780-1781.)—A process is described for the detection and estimation of the amino-acids resulting from the hydrolysis of proteins depending on the conversion of the amino-acids into their betaine compounds by the exhaustive methylation of the hydrolysed mixture. The method was carried out as follows in the case of spongin: 94 grms. of spongin were hydrolysed with hydrochloric acid, the mixture precipitated with mercuric chloride, the precipitate decomposed with hydrogen sulphide, and the filtrate from the mercuric sulphide precipitate was evaporated to dryness, and the residue dissolved in methyl alcohol containing potassium hydroxide. The solution was then cooled to 0° C., and dimethyl sulphate was added in small portions at a time. The alkaline mixture, after standing for twelve hours, was heated for a short time on a water-bath, then acidified with hydrochloric acid, and concentrated. After precipitating inorganic salts by the addition of absolute alcohol, the solution was evaporated to a syrupy consistence, acidified with hydrochloric acid, a large excess of mercuric chloride solution was added, and allowed to stand for twenty-four hours. The crystalline precipitate obtained was then collected, decomposed with hydrogen sulphide, the solution evaporated to a syrup, and precipitated with platinum chloride in alcoholic solution. The precipitate consisted of the platinum salt of *n*-methyl hygrinic acid. The yield was 2·982 grms., consequently 4·5 per cent. of the spongin consists of pyrollidine carboxylic acid. The gold salt had the composition: $C_7H_{14}NO_2.AuCl_4$. The process was also applied to extract of ergot. This was acidified with sulphuric acid, and precipitated with phosphotungstic acid, and the solution, after the removal of phosphotungstic acid, leucine, etc., was evaporated to a syrup and treated with dimethyl sulphate; 0·45 grm. of betaine chloride was isolated and about 0·22 grm. of glycocoll, corresponding with about 4·5 per cent. of the total nitrogen present. It was also found that tryptophane cannot be methylated by means of methyl iodide. W. P. S.

Herzig and Meyer's Reaction Applied to Proteins and Amino-Acids.

J. H. Burn. (*Biochem. J.*, 1914, **8**, 154-157.)—This reaction consists in heating the substance with hydriodic acid, so that the methyl groups leave the nitrogen to form methyl iodide, and is carried out similarly to the Zeisel method of estimating methoxy groups. The decomposition of methylamino groups requires a temperature of 230° C., whereas methoxy groups are decomposed at 130° C. Glycerol and glycerides give rise to isopropyl iodide and to a silver iodide precipitate. It was found that commercial gelatin, Hammarsten's casein freed from fat, and a dried pancreatic digest of protein freed from fat, gave positive results. The phosphotungstic precipitate from the digest and the filtrate from the precipitate and the amino-acids glycine, alanine, leucine, valine and histidine, also gave positive results, while tyrosine and tryptophane gave negative results. It is possible that methylamino groups exist in protein, but this reaction does not afford a reliable test for their presence. E. W.

Titration of Milk with Alcohol at Different Degrees of Concentration.

F. Lohnis. (*Molkerei Zeitung*, 1914, **9**, 153-155; through *Bull. Agric. Intell. and Plant Diseases*, 1914, **4**, 534-535.)—The coagulation of inferior or bacterially contaminated milk, when mixed with its own volume of 68 to 70 per cent. alcohol (by volume), though a useful test, is unreliable at times, and the author has obtained better results with stronger alcohol. Two c.c. of milk is the best quantity to use, the alcohol being allowed to fall in drop by drop, and the formation of the precipitate is best seen by tilting the beaker against a black background. The "alcohol number" is the volume required to cause a precipitate in 2 c.c. of milk, the following generalisation being the result of work on over seventy samples:

Low bacterial content:	upwards of 4 c.c.	of alcohol	(80 per cent. by volume)
Average ,, ,,	2 to 4 c.c.	,, ,,	(80 ,, ,,)
High ,, ,,	less than 2 c.c.	,, ,,	(80 ,, ,,)
Very high ,, ,,	,, ,, ,,	,, ,,	(70 ,, ,,)

These values show no noticeable difference from those obtained when using the methylene blue test, but the alcohol method is to be preferred, as it allows a low bacterial content (less than 100,000) to be recognised and the keeping qualities of the milk to be determined. In winter the alcohol numbers have always been found very low, and the variation sometimes met with from day to day, when working on the same sample, appears to depend chiefly on the weather and the air temperature. As a rule the first milk gives higher alcohol numbers than that obtained towards the end. H. F. E. H.

Assay of the Amylolytic Diastase of Pancreas. P. Macquaire. (*Comptes rend.*, 1914, **158**, 1289-1291.)—An average sample of fresh pancreas from a large number of pigs was separated from the fat and reduced to a pulp. Of this preparation, quantities of 0·05 grm. each were caused to act on a series of starch pastes containing from 1 to 6 grms. of potato starch at a temperature of 55° C. for exactly one hour. The pastes were made at a concentration of 5 per cent., with the exception of the last, when it was 6 per cent. Similar series of tests were made with increasing quantities of pancreas—viz., 0·10, 0·15, 0·20, and 0·25 grm.—and the quantities of reducing sugars estimated after one hour at 55° C. The tests showed that the quantity of starch converted into reducing sugar by 0·05 grm. of pancreas acting on 1 grm. of starch under the conditions indicated was 44 per cent. When five times the quantity of ferment acted on five times the quantity of starch, the conversion fell to 38·4 per cent. Thus the coefficient of transformation decreases as the quantity of substance to be converted is increased. On the other hand, if the quantity of ferment be increased while that of the starch is kept constant at 1 grm., the degree of conversion does not exceed 45 per cent.; and when the starch is increased to 5 grms., the conversion always remains at 38·4 per cent., in spite of the excess of ferment. According to the official prescriptions for the assay of pancreatin, the conversion produced in 100 grms. of paste containing 5 grms. of starch must be equivalent to at least 2 grms. of reducing sugar, whereas the author's experiments show that not more than 1·92 grms. of sugar can be produced under these conditions. In order to obtain the amount of sugar specified, it would be necessary to work with a paste containing 6 grms. of starch in 100 grms. J. F. B.

Measuring the Capillary Lift of Soils. C. J. Lynde and H. A. Dupré. (*J. Amer. Soc. Agronom.*, 1913, **5**, 107-116; through *Bull. Agric. Intell. and Plant Diseases*, 1914, **4**, 457.)—Small glass funnels, 4 cm. in diameter, are fitted with cotton-cloth filters cut in the form of a circle 2 cm. in diameter and folded like a filter-paper. Soil samples are boiled in water, and a little of the hot mixture is poured on the filter, and the funnel centrifuged, the process being repeated until the level of the soil in the funnel is well above the top of the filter-cloth. The lower end of each funnel is then connected with a vertical capillary tube filled with water, the lower end dipping under mercury. As water evaporates from the funnel the rise of mercury is noted. The theoretical capillary lift was calculated for the different soil fractions, and in all cases except the clay fraction the observed values fell between the limits of the calculated values. In the case of clay and some other soils the calculated values surpassed 34 feet of water, but in no case was there an observed rise of this magnitude, which is equivalent to one atmosphere. It was found that the limiting factor was the atmospheric pressure, for by enclosing the whole apparatus in a glass case, and varying the air-pressure, a corresponding rise or fall of the capillary lift was observed. The method is rapid, twenty hours being sufficient to get a lift equivalent to 30 feet of water. H. F. E. H.

Volumetric Method for the Estimation of Ethereal and Inorganic Sulphates in Urine. O. Rosenheim and J. C. Drummond. (*Biochem. J.*, 1914, **8**, 143-152.)—The sulphates are precipitated as benzidine sulphate, which is estimated by titration with $\frac{N}{10}$ potassium hydroxide. For the estimation of inorganic sulphates, 25 c.c. of urine are made distinctly acid to Congo-red paper with dilute hydrochloric acid (1 : 4); 100 c.c. of the benzidine solution (4 grms. benzidine and 5 c.c. hydrochloric acid (sp. gr. 1·19) diluted to 2 litres) are then added, and the precipitate, after being allowed to settle for ten minutes, is filtered and washed with 10 to 20 c.c. of water saturated with benzidine sulphate. The precipitate and filter-paper are transferred to the original precipitation-flask with about 50 c.c. water, and titrated hot with $\frac{N}{10}$ potassium hydroxide, using phenolphthaleïn as indicator. It is important that the precipitate should be finely suspended in water before titration, and for this reason the precipitate should not be allowed to dry on the filter. It is necessary to employ a stronger phenolphthaleïn solution (a few drops of a saturated alcoholic solution) than usual, as it is adsorbed to a considerable extent by the filter-paper. For the estimation of the total sulphates (inorganic and ethereal), 25 c.c. of urine are treated with 2 to 2·5 c.c. of hydrochloric acid (1 : 4) and 20 c.c. water, and the mixture boiled for fifteen to twenty minutes. After cooling, the liquid is precipitated with benzidine solution, and the precipitate treated as described above. The quantity of acid recommended is sufficient for complete hydrolysis, but if more acid is used it is necessary, before precipitation, to neutralise and reacidify until acid to Congo red. The accuracy of the method was tested by comparison with Folin's gravimetric barium method. Most of the results agreed; the largest difference amounted to −0·0025 per cent. in favour of the barium method, and to +0·001 per cent. in favour of the benzidine method. E. W.

ORGANIC ANALYSIS.

Specific Reaction of Ethyl Alcohol. A. Toninelli. (*Ann. Chim. anal. Appl.*, 1914, **19**, 169-170.)—Two c.c. of the distillate under examination are shaken in a stoppered tube with 2 c.c. of a solution of 12 grms. of iodine in 100 c.c. of ether, and then, after two minutes, with 4 c.c. of potassium hydroxide solution (40 grms. per 100 c.c.) until decolourised. It is then tested with 2 c.c. of a solution of 1·5 grms. of pure dinitrotoluene in 200 c.c. of a mixture of 1 part of carbon disulphide and 2 parts of ether. In the presence of ethyl alcohol (3 per cent. and over) the upper layer, which separates after the shaking, assumes an orange colour, which gradually fades, and then becomes red. Methyl alcohol, acetone, and aldehyde do not interfere with the reaction, but similar colorations are given by certain higher alcohols. If the presence of such be suspected, from 25 to 100 c.c. of the sample are repeatedly shaken with twice the volume of a 5 per cent. solution of alum, and a little benzene or petroleum spirit. The aqueous layer is drawn off and distilled in a fractionating still, and the portions distilling between 60° and 80° C. tested as described. If insufficient distillate is obtained, 3 c.c. of pure acetone are added, and the distillation repeated. C. A. M.

Solvents for Coal. L. Vignon. (*Comptes rend.*, 1914, **158**, 1421.)—Experiments with various solvents showed that the quantity of matter extracted by aniline depends upon the content of volatile matter in the coals, and affords a basis for distinguishing between them. The extract from a gas coal, after precipitation with acid, is richer in hydrogen, and gives less ash than the insoluble portion, and yields a coherent, porous coke instead of a pulverulent one. O. E. M.

Use of Diphenylbenzidine in Place of Diphenylamine as a Reagent for Oxidising Substances. Marqueyrol and Muraour. (*Ann. Chim. anal. Appl.*, 1914, **19**, 174-176.)—The blue coloration given by diphenylamine with oxidising agents is due to the formation of a quinone salt of diphenylbenzidine. The latter substance is therefore a much more sensitive reagent, and will give a blue coloration with traces of oxidising substances insufficient to do more than convert diphenylamine into diphenylbenzidine. The reagent may be prepared by adding to a cold mixture of 50 c.c. of sulphuric acid (sp. gr. 1·84) and 150 c.c. of water a solution of 8 grms. of diphenylamine in 20 c.c. of acetic acid, and adding, with continual stirring, a solution of 4·7 grms. of sodium bichromate in 200 c.c. of water and 29 c.c. of sulphuric acid (1 : 4). After five minutes the liquid is added to a solution of 100 c.c. of sodium bisulphite (sp. gr. 1·23) and 400 c.c. of water, and the mixture heated on the water-bath until the green precipitate turns brown, after which it is cooled and filtered, the precipitate washed with water and alcohol, dried, and dissolved in boiling toluene, and the solution filtered hot and set aside for the diphenylbenzidine to crystallise. C. A. M.

Method of Estimating Formic Acid. V. Hottenroth. (*Chem. Zeit.*, 1914, **38**, 598.)—Formic acid reacts with acid anhydrides, yielding the corresponding acid accompanied by an evolution of carbon monoxide, the reaction proceeding according to the equation:

$$CH_3COO.COCH_3 + HCOOH = 2CH_3COOH + CO.$$

A similar reaction takes place with acid chlorides, but in this case HCl is liberated. The reaction takes place only when the mixture is warmed or in the presence of a contact-substance—*e.g.*, a trace of concentrated sulphuric acid. For instance, if a mixture of formic acid and acetic anhydride is placed in a flask and a drop of concentrated sulphuric acid is added, a violent evolution of carbon monoxide immediately takes place. By allowing the reaction to take place in an apparatus where the volume of the carbon monoxide may be measured, the method affords a means of estimating formic acid. It may also be used for the detection of traces of formic acid. The formic acid may, for the sake of convenience, be dissolved in a homologous acid—glacial acetic acid, for instance—before being added to the acetic anhydride, and, in order that traces of water may be eliminated, the mixture is allowed to stand for a short time before the sulphuric acid is introduced. W. P. S.

"Vacuum Distillation" Method for the Detection of Formic Acid. T. Merl. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1914, **27**, 733-743.)—Experiments carried out by the author show that small quantities of formic acid can be separated from solutions also containing sugars, organic acids, etc., by distillation under low pressure, and that under these conditions there is no tendency for the continuous formation of traces of formic acid from the distillation materials. Distillation under reduced pressure in the presence of steam yields less trustworthy results. The formic acid obtained in the distillates was estimated by the mercuric chloride method (*cf.* ANALYST, 1913, **38**, 378). W. P. S.

Separation of Hydrocarbons by Means of Liquid Sulphur Dioxide. L. Edeleanu. (*Petroleum*, 1914, **9**, 862-864; through *Chem. Zentralbl.*, 1914, I., 1785.) —The aromatic and cyclic unsaturated hydrocarbons dissolve in liquid sulphur dioxide, whereas the paraffins and naphthenes are almost insoluble. A measured quantity (50 c.c.) of the oil to be examined is transferred to a 200 c.c. burette, which is expanded into a bulb at the top and also at the bottom between the graduated stem and the cock. The burette is placed in a metal cylinder containing kerosine, and this cylinder is surrounded by a freezing mixture. About 100 c.c. of liquid sulphur dioxide is transferred to a 200 c.c. cylinder with cock near the bottom, and this cylinder is also placed in a cooled oil-bath. When both the oil to be examined and the sulphur dioxide have cooled at least to − 12° C., sulphur dioxide is run into the burette until shaking no longer causes the lower layer to disappear. About one-third of the sulphur dioxide remaining in the cylinder is run into the burette, the contents of which are shaken and cooled once more to − 12° C. The lower layer is then drawn off into a Dewar vessel. The sulphur dioxide remaining in the cylinder is transferred to the burette, the contents of which are shaken and cooled as before, and the lower layer is again run off into the Dewar vessel. The undissolved saturated hydrocarbons remain in the burette. With distillates of high boiling-point (lubricating oil, gas oil) it suffices to evaporate the dissolved sulphur dioxide, and weigh or measure the residual saturated hydrocarbons. With benzine and even with kerosine this is not permissible. In such cases the contents of the burette are allowed to flow into a cooled solution of alkali, contained in a flask with graduated neck and side tube in the shoulder. Water is finally introduced through this side tube until the oil lies wholly within the graduated part of the neck. The aromatic and cyclic hydrocarbons are estimated by difference. G. C. J.

Determination of the Softening Point of Pitch. Klinger. (*Chem. Ind.*, 1914, **37**, 220; through *Chem. Zentralbl.*, 1914, I., 1857-1858.)—A quantity of the pitch is melted in a beaker in an oil-bath at a temperature of 150° C., so that the layer of melted pitch on the bottom of the beaker is about 7 mm. in depth. A glass tube, 10 cm. long and 6 mm. internal diameter, is then inserted in the beaker, and after one minute the upper end of the tube is closed with the finger, and the tube, now containing a layer of pitch, is removed and placed upright on a cold glass plate. When cold, the solid pitch is removed from the outside of the tube, and 5 grms. of mercury are poured into the upper end of the tube so as to form a layer on the surface of the solid pitch contained in the lower part of the tube. The latter is now immersed in a beaker containing water at 40° C., this beaker is placed in a larger beaker also containing water at the same temperature, and the temperature of the water in the outer beaker is raised at the rate of about 1° C. per minute. The temperature (ascertained by a thermometer in the inner beaker) at which the mercury falls through the layer of pitch is taken as the softening-point of the latter (*cf.* ANALYST, 1914, 264). W. P. S.

Dry and Wet Strengths of Paper and Paper Yarns. C. Beadle and H. P. Stevens. (*Chem. News*, 1914, **109**, 242-244.)—After thorough steeping in water, the great majority of commercial papers do not possess sufficient strength to show any result on an ordinary tensile testing machine. Two Swedish "kraft" papers of the type used for twisting paper yarns showed breaking lengths, when tested dry, of 9·7 and 9·6 km., but when tested wet the respective values were 0 and 0·58 km. Similar paper made from *Hedychium coronarium* showed in dry state 10·03 km. and 1·58 km. when thoroughly wetted, this superiority in wet strength being due to the parchment-like qualities of the *Hedychium* paper. When the papers are twisted into yarn, a notable decrease in dry strength is recorded as the result of the treatment; thus the Swedish papers yielded yarns showing 6·89 km. and the *Hedychium* paper 7·37 km. breaking length. On the other hand, a very considerable increase in the wet strength is produced, the Swedish paper yarn showing 4·18 km. and the *Hedychium* paper yarn 4·90 km. when fully wetted. The decrease in tensile strength of a paper yarn when wetted is therefore about 36 per cent. These observations were made with yarns twisted from finished paper damped at the time of twisting, according to modern practice. Yarns twisted from partly manufactured moist paper by the older processes are generally stronger than the untwisted paper, but the absolute strength values of the modern yarns are better than those of the older ones. Paper yarns are generally prepared from papers of a substance of 40 to 44 grms. per sq. m., the count of the yarn being varied by varying the width of the twisted strips; the number of twists to the inch also affects the thickness and weight of the yarn. J. F. B.

Viscosity of Rubber Solutions. R. Gaunt. (*J. Soc. Chem. Ind.*, 1914, **33**, 446.)—The behaviour of five samples of rubber to benzene, ether, petroleum spirit, toluene, xylene, and chloroform was investigated, and the viscosity of the solutions determined. The results were compared by plotting the logarithm of the viscosity against the concentration, the inclination to the abscissa of the straight lines thus obtained giving numerical values for the viscosities. The fall in viscosity on exposure to light and to high temperature was studied, and for a solution maintained at 100° C. away from light was found to follow the law $x = a + b \log. t$, x being the diminution in time t, a the diminution in the first time unit (thirty minutes), and b the increment of diminution with time. The rate of fall was greater the higher the concentration, and greater in xylene than in benzene solution; the *Hevea* rubbers showed a higher rate than the other species. O. E. M.

Estimation of Semicarbazide by its Interaction with Halogens and Halogen Oxyacids. R. L. Datta.

(*J. Amer. Chem. Soc.*, 1914, **36**, 1014-1017.)—When a solution of semicarbazide hydrochloride is treated with powdered potassium chlorate and hydrochloric acid, it is decomposed with the liberation of exactly two-thirds of its nitrogen in the elementary condition, the remaining third being fixed as ammonium chloride. As carbamide, under similar conditions, is unacted on, the reaction affords a means of estimating semicarbazide in presence of carbamide. The reaction is conveniently brought about in a nitrometer. The other gaseous products are carbon dioxide and chlorine, and the latter must be washed down before introducing alkali hydroxide to absorb the former, as otherwise hypochlorite would be formed. Hypochlorite (or hypobromite) decomposes semicarbazide, like carbamide itself, with the liberation of the whole of the nitrogen as such.

When potassium bromate is substituted for potassium chlorate, nearly three-quarters of the nitrogen in semicarbazide is liberated as such, and in concentrated solutions the reaction does not require the addition of hydrochloric acid to start it. The action of iodate is still stronger, exactly three-quarters of the nitrogen in semicarbazide being liberated even from very dilute solutions, and without the help of any acid. G. C. J.

Inversion of Cane Sugar by Acids in Water-Alcohol Solutions. G. J. Burrows.

(*J. Chem. Soc.*, 1914, **105**, 1260-1270.)—The author has measured the rates of inversion of cane sugar by hydrochloric and sulphuric acids in mixtures of water and ethyl alcohol up to 75 per cent. of alcohol, and finds that in all cases a minimum velocity is associated with a solution containing 45 to 50 per cent. of alcohol. The velocity of inversion in such mixtures appears to be a function of the fluidity of the solution. By considering the velocity to depend on the concentration and mobility of the hydrogen ions as determined by conductivity, the catalytic activity of the ions is shown to be greater in alcoholic than in aqueous solutions. The relative activities of $\frac{N}{2}$ hydrochloric and sulphuric acids are shown to vary with the solvent; thus in 75 per cent. alcohol hydrochloric acid has a greater strength relative to sulphuric acid than in water. It would be expected that the amount of dissociation of the sulphuric acid would be reduced to a greater extent by alcohol than that of the stronger acid, hydrochloric acid. All alcohol strengths given are per cent. by volume. H. F. E. H.

Note by Abstractor.—There is no evidence in the paper that any correction has been made for the effect of the alcohol on polarimeter readings of the lævulose, although the author is aware that such effect is considerable. The effect of birotation is also assumed to be negligible twenty minutes after the addition of the acid to the sugar-alcohol solution at the temperature of the experiments (25° C.).

Detection and Estimation of Lævulose in Presence of Other Sugars. F. Pinoff and K. Gude.

(*Chem. Zeit.*, 1914, **38**, 625-626.)—The ammonium molybdate reaction for the detection of lævulose in presence of other sugars is more reliable when modified in the following manner: Six grms. of powdered ammonium molybdate are dissolved in 5 c.c. of boiling water, the solution is cooled to 40° C. and added to 5 c.c. of the solution containing lævulose. This solution must contain no free acid, otherwise sugars other than lævulose will respond to the test. The addition is made in such a way that the whole of the molybdate, part of which may have separated through cooling, is contained in the 10 c.c. of mixed solutions. The 5 c.c. of the sugar solution should contain not more than 1·0 and not less than 0·03 grm. of sugar. The mixture is heated at 40° C. in the water-bath for fifteen minutes, and gives a blue coloration if lævulose be present. If dextrose is present to the extent of over 1·0 grm. in 5 c.c., it will itself give a blue coloration equivalent to that shown by 0·03 grm. of lævulose; hence the necessity for limiting the concentration. As a quantitative colorimetric method the above reaction does not give satisfactory results, but the following method, based on the formation of a blue colouring matter with diphenylamine, showing an absorption band corresponding to $\mu = 500\text{-}550$, is recommended: 2·5 c.c. of the lævulose solution, 2·5 c.c. of 96 per cent. alcohol, 10 c.c. of a mixture of 750 c.c. of 96 per cent. alcohol, and 200 c.c. of concentrated sulphuric acid, and 0·1 grm. of diphenylamine, are heated together at 70° C. under a reflux condenser for fifteen minutes, then boiled for three minutes, and immediately cooled. The blue solution is transferred to a Hehner's colorimeter glass and diluted with sufficient alcohol to produce a light blue liquid, which is then examined spectroscopically. Dilution is continued until the absorption band is only just perceptible. In order to calculate the percentage of lævulose in the solution, the degree of dilution (*i.e.*, the number of volumes of alcohol added to the blue liquid) + 1, is multiplied by the factor 0·036, which represents the limiting concentration of lævulose, giving a perceptible band. The concentration of the sugar solution should be approximately 1 per cent., and should not exceed 10 per cent., of total sugars. A 25 per cent. solution of dextrose will give a blue coloration equivalent to a 0·1 per cent. solution of lævulose. J. F. B.

Estimation of Lactose by the Copper-Iodide Method. S. W. Cole. (*Biochem. J.*, 1914, **8**, 134-143.)—A volume of a copper sulphate solution of known strength is treated with solutions of alkaline tartrate and the sugar, and the mixture heated under standardised conditions for a known time. After filtering, the excess of copper is determined by acidifying with sulphuric acid, adding potassium iodide, and titrating with sodium thiosulphate, these operations being carried out under prescribed conditions of temperature, dilution, and acidity. The curves representing the amounts of copper reduced for varying quantities of lactose are almost linear.

E. W.

INORGANIC ANALYSIS.

Estimation of Carbon Dioxide in Air. W. M. Doherty. (*Report of the Australasian Association for the Advancement of Science*, 1913, **14**, 100-102.)—A series of about ten 100 c.c. flasks well stoppered and filled with water free from carbon dioxide are emptied at the place where the air sample is required and restoppered. Into each flask is then run a volume of standard sodium carbonate solution, coloured with phenolphthaleïn (1 c.c. equals 0·01 c.c. of carbon dioxide), beginning at about 4 c.c. and increasing by 1 c.c. increments, until a limit is reached beyond which it is unnecessary to go. The end-point lies between the last decolourised flask and the next in the series which just retains its pinkness. The solution of sodium carbonate referred to above is made by diluting to a litre 10 c.c. of a solution which contains 0·4748 grm. sodium carbonate per 100 c.c.; and must be suitably protected from the air. When 10 c.c. are diluted to a litre it is ready for use as above, and should be shaken with the air sample continuously for twenty minutes.

H. F. E. H.

Separation of Beryllium from Uranium. M. Wunder and P. Wenger. (*Zeitsch. anal. Chem.*, 1914, **53**, 371-374.)—The method proposed depends on the precipitation of uranium from a solution of its chloride by means of hydrogen peroxide; beryllium is not precipitated by this reagent. A neutral or feebly acid solution of the two chlorides is treated with hydrogen peroxide solution and heated on a water-bath until oxygen is evolved freely; the yellow precipitate is then collected on a filter, washed with dilute hydrogen peroxide solution, or dilute ammonium nitrate solution, ignited in a platinum crucible, and weighed. The ignited precipitate consists of U_3O_8. After the excess of hydrogen peroxide has been expelled from the filtrate by heating, the beryllium may be precipitated with ammonia, ignited, and weighed as BeO. When the quantity of beryllium is large as compared with that of the uranium, a double precipitation with hydrogen peroxide is necessary for the complete separation. The nitrates of the two metals may be separated in the same way.

W. P. S.

Estimation of Colloids in Effluents and the Use of the Interferometer in Water Analysis. R. Marc and K. Sack. (*Kolloidchem. Beihefte*, 1914, **5**, 375-410; through *Chem. Zentralbl.*, 1914, I., 1849.)—The method described depends on the fact that the colloids present in an effluent are absorbed by finely divided insoluble inorganic substances, such as barium sulphate, whilst the crystalloids are not affected; further, the alteration in the concentration of the colloids in the effluent may be determined by means of the Zeiss interferometer (*cf.* ANALYST, 1912, **37**, 382). The authors find that about one-half of the organic substances in ordinary household waste water are present in a colloidal form; these colloids are rapidly decomposed during the natural purification of the waste water.

W. P. S.

Colorimetric Estimation of Small Quantities of Hydrogen Sulphide. W. Mecklenburg and F. Rosenkränzer. (*Zeitsch. anorg. Chem.*, 1914, **86**, 143-153.)—The principle of the method depends on the production of methylene blue when dimethylparaphenylenediamine sulphate, ferric chloride, and hydrochloric acid are added to aqueous solutions of hydrogen sulphide. The sensitiveness of this reaction as a qualitative test has long been known, and previous attempts have been made to make it the basis of a quantitative method (*e.g.*, W. G. Lindsay, *School of Mines Quarterly*, 1901, **23**, 24). The authors find that it will detect 1 part of hydrogen sulphide in 100 million parts of water, and that proportions ranging from 0·05 to

3 parts per million can be estimated with an error not exceeding 4 per cent. To attain this degree of accuracy, however, it is found that close attention must be given to the exact experimental conditions under which the colour is produced. To 500 c.c. of the water to be tested the authors add in quick succession, and always in this order, 10 c.c. of concentrated hydrochloric acid, 25 mgrms. of dimethylparaphenylenediamine sulphate, and 2·5 c.c. of a solution containing 27 grms. of crystallised ferric chloride per litre. The coloration increases for some time, but often reaches a maximum and then falls somewhat before becoming constant. A constant value is attained in about three hours, after which the colour intensity remains unchanged for at least four months, if the solution is not unduly exposed to light. Comparison is made with standard solutions made up in a similar manner with dilute solutions of hydrogen sulphide standardised iodimetrically. Comparison cannot be made with solutions made by dissolving pure methylene blue, as these solutions are pure blue, whereas those obtained in this test, especially the paler ones, are greenish. Comparatively few standards are necessary, as the intensity of coloration is strictly proportional to the concentration of hydrogen sulphide originally present.

It is not necessary to use the exact quantities of the several reagents mentioned above, but it is important to use the same quantities in an actual assay as were used in preparing the standards. The quantities named suffice for the estimation of as much as 1·5 mgrm. of hydrogen sulphide in 500 c.c. If waters containing much less hydrogen sulphide than this are to be tested, 10 mgrms. of dimethylparaphenylenediamine sulphate and 1 c.c. of ferric chloride solution may be used; but the resulting colours will be paler than if larger quantities of the reagents were used, and must be compared with standards prepared in a similar way. Variation of the concentration of hydrochloric acid between 5 and 15 c.c. per 500 c.c. is without effect, as is the presence of sulphuric acid up to 1·25 per cent., of sodium chloride up to 6 per cent., and of zinc chloride up to 2 per cent. The temperature of the liquid whilst the reaction is in progress is not without effect on the ultimate colour intensity, and all work should be conducted at some standard temperature ±2° C. Once the colour has developed, it is not affected by ordinary changes of temperature. G. C. J.

Colorimetric Estimation of Cobalt, Nickel, Iron, and Copper. C. Hüttner. (*Zeitsch. anorg. Chem.*, 1914, **86**, 341-357.)—In strong hydrochloric acid solution, cobalt chloride has a dark blue colour, nickel chloride an intense yellow colour, and small quantities of cobalt can be estimated in nickel alloys with sufficient exactness as follows, provided copper and iron are absent: Of the alloy, 0·2 grm. is dissolved in nitric acid, and the solution evaporated to dryness three times with hydrochloric acid. The residue is dissolved in 3 drops of water and a little hydrochloric acid, and the solution is diluted to 100 c.c. with hydrochloric acid of 1·19 sp. gr. and transferred to a colorimeter cylinder. A similar cylinder is charged with 95 c.c. of a 0·2 per cent. solution of nickel in hydrochloric acid of 1·19 sp. gr., and a 0·2 per cent. solution of cobalt in acid of the same strength is added from a burette until a match is obtained. The second cylinder is then filled to the 100 c.c. mark with the standard nickel solution, and if necessary one more drop of cobalt solution is added to restore the equality of tint. Copper, iron, chlorine, and nitric acid must be absent. Acid of the same strength must be used in all experiments.

It is well known that the intensity of coloration of hydrochloric acid solutions of ferric chloride is greatly influenced by the concentration of acid. The maximum coloration is obtained with acid of 1·14 sp. gr. Provided, however, that the concentration of acid is kept approximately the same in all experiments, the coloration affords a convenient means of estimating small quantities of iron. Though not available for estimating the minute traces that can be estimated by means of thiocyanate, for somewhat larger quantities the method is preferable, as the only factor influencing the colour, apart from the quantity of iron, is the concentration of acid, and this is easily controlled. Comparison is made with a standard solution containing 1 part of iron in 20,000 parts of hydrochloric acid of the same strength as is used in the actual assays. The author prefers to use acid of 1·19 sp. gr., although somewhat weaker acid gives a more intense coloration. Copper must be absent. Cobalt and nickel in small quantity do not interfere, but if large quantities are present in the material under examination, the iron must be separated as hydroxide and redissolved. The small quantity of nickel which tends to be co-precipitated with the ferric hydroxide is without influence on the results. Chlorine does not interfere, but nitrates must be absent.

In absence of iron, copper may be estimated in a manner exactly similar to that described for iron. The maximum colour is attained in this case by using acid of 1·19 sp. gr. Chlorine does not interfere, but nitrates must be absent. Iron must not be present, nor large quantities of nickel or cobalt. G. C. J.

Colorimetric Estimation of Nickel. V. Lindt. (*Zeitsch. anal. Chem.*, 1914, **53**, 165-175.)—The method depends on the depth of colour produced when potassium thiocarbonate is added to an ammoniacal solution containing nickel. Among the other metals which may be present in an ammoniacal solution, only copper, manganese, cobalt (-ous), and to a less extent zinc, interfere. Copper must be separated as sulphide, but manganese can be removed with iron, and cobalt salts oxidised to cobaltic salts which do not interfere, by making the ammonia precipitation in presence of bromine or hydrogen peroxide. Zinc does not develop a coloration with the reagent, but gives rise to a white precipitate. This precipitate forms so slowly, however, that the author has found no difficulty in estimating nickel in presence of much zinc. It is suggested that any possible difficulty due to this precipitate, in circumstances such that the estimation could not be carried out sufficiently fast to prevent its formation, might be overcome by making comparison with standards containing zinc as well as nickel.

To 20 c.c. of the ammoniacal solution of nickel 0·5 c.c. of a 4 per cent. solution of potassium thiocarbonate is added, and the depth of colour of the solution is compared with that of standards containing known amounts of nickel, and prepared simultaneously. The solution of potassium thiocarbonate should not be more than a week old. Comparison is most easily made between solutions containing from 0·3 to 2 mgrms. nickel in 20 c.c. Between these concentrations the colour developed is a more or less intense red, and comparatively small differences in concentration can be readily detected. In concentrations above 2 mgrms. in 20 c.c. the increase in colour intensity with increasing concentration is less marked, whilst in concentrations below 0·2 mgrm. per 20 c.c. the colour developed is not red, but yellow, and its intensity is sensibly modified by factors other than the concentration of nickel.

G. C. J.

Electrolytic Reduction of Iron for Permanganate Titration. H. C. Allen. (*J. Amer. Chem. Soc.*, 1914, **36**, 937-949.)—It is possible to get practically complete reduction of ferric iron by using a large platinum cathode and a small platinum wire anode, if the concentration of sulphate is small. By using a soluble anode and platinum cathode, complete reduction is possible with wide range of acidity, but with low acidity some of the iron may be deposited on the cathode with high-current density. Chlorides must be absent with platinum anodes, but with soluble anodes may be present, unless too great current density is employed. By using a short-circuited cell with platinum gauze cathode and a zinc anode, complete reduction may be obtained with wide range of acidity.

The most practical method of reduction in most cases dispenses with an outside source of current, and makes use of a short-circuited cell with copper gauze cathode and amalgamated zinc rod anode, the cathode being rotated. The concentration of acid must be at least 10 c.c. concentrated sulphuric acid per 175 c.c. Under these conditions, 0·12 grm. iron may be reduced at room temperature in eight minutes with a zinc efficiency of 70 per cent. Chlorides do not interfere.

Increase of temperature in every case increases the rate of reduction.

G. C. J.

Analysis of Higher Oxides of Lead. J. Milbauer and B. Pivnička. (*Zeitsch. anal. Chem.*, 1914, **53**, 345-359.)—A systematic study of the iodimetric methods proposed for the analysis of red lead and lead peroxide shows that, whilst the objections of Chwala and Colle to these methods are well founded, it is possible to obtain correct results under certain conditions. The modified method described is as follows: Five grms. of sodium acetate, 0·5 grm. of potassium iodide, 100 c.c. of water, and 5 c.c. of glacial acetic acid, are placed in a flask, the solution is cooled as low as possible, and a weighed quantity of the lead oxide is added (0·5 grm. of red lead, or 0·2 grm. of lead peroxide); the liberated iodine is then titrated with $\frac{N}{100}$ thiosulphate solution, the mixture being shaken continuously during the titration. The thiosulphate solution must be standardised immediately before use.

W. P. S.

Note on Lux's Method for the Estimation of Lead Peroxide. A. Ipiens. (*Zeitsch. anal. Chem.*, 1914, **53**, 261-265.)—In the titration of oxalic acid with permanganate in presence of lead nitrate and nitric acid, the end-point of the titration is reached very slowly, and several small additions of permanganate are necessary before the pink colour becomes permanent for five minutes. Even then it is found that with $\frac{N}{10}$ solutions—made perfectly equivalent when used alone—a difference appears in presence of lead nitrate, so that 25 c.c. of oxalic solution correspond only to 24·4-24·6 c.c. of the permanganate. The author has obtained far better results with a liquid containing oxalic acid, nitric acid, and lead nitrate, by neutralising the acids with sodium or potassium hydroxide and then acidifying with sulphuric acid. In this way 25 c.c. of $\frac{N}{10}$ oxalic acid solution required 24·8 or 24·75 c.c. of permanganate, and the end-point was reached in a perfectly normal manner. Using this method, the author has investigated the electrolysis of lead nitrate solutions strongly acidified with nitric acid, in which case the lead is precipitated at the anode as peroxide. The precipitate was weighed after drying at 220° to 230° C., and was also analysed volumetrically by titration after reduction with oxalic acid as described above. The volumetric results were always slightly lower than the gravimetric values for the lead peroxide, so that Hollard's suggestion that the precipitate includes some of a higher oxide than PbO_2 is not confirmed. The difference in the results may be partly due to the fixation of small quantities of water by the lead peroxide, and partly to the presence of traces of lead nitrate, which could, moreover, be detected in the precipitate. J. F. B.

Estimation of Nitrogen in Mixtures of Calcium Nitrate and Calcium Cyanamide. A. Stutzer. (*Chem. Zeit.*, 1914, **38**, 597.)—Although the usual methods for the estimation of nitric and cyanamide nitrogen yield correct results when applied separately to nitrates and cyanamides, the figure for total nitrogen present in mixtures of these compounds is usually too low. Trustworthy results may, however, be obtained by estimating the nitric nitrogen by Schloesing's method, and then estimating the cyanamide nitrogen in another portion of the sample by Kjeldahl's method after all the nitric nitrogen has been expelled by heating the substance with ferrous chloride and hydrochloric acid. W. P. S.

Estimation of Phosphates in Soil Extracts. J. A. Prescott. (*J. Agric. Sci.*, 1914, **6**, 111-120.)—After critically examining various methods, the author finally adopted the following: The reagents required are—(1) Ammonium nitrate, 500 grms. in a litre; (2) ammonium molybdate solution, 150 grms. dissolved in a litre of water and poured into a litre of nitric acid (sp. gr. 1·2); (3) 2 per cent. sodium nitrate. A measured volume of soil extract containing 5 to 10 mgrms. of P_2O_5 is evaporated to dryness, the residue being ignited at a dull red heat for fifteen minutes, and then taken up with 50 c.c. of 10 per cent. sulphuric acid, and digested for half an hour on a sand-bath. The extract is diluted if necessary, filtered, and the residue washed with hot water, filtrate and washings amounting to 110 c.c. This procedure is found to extract all the phosphate when the amount of the original solution is not more than 100 c.c. in the case of a hydrochloric acid extract. For soil extracts containing much silica (such as extracts made with 1 per cent. citric acid) it is necessary to heat the residue from the evaporation for two hours at 120° to 160° C., otherwise the silica subsequently interferes. To the solution prepared as above are added 25 c.c. of the concentrated ammonium nitrate, and the mixture brought to 55° C. Twenty-five c.c. of the ammonium molybdate solution previously brought to the same temperature are added, and the mixture stirred, allowed to cool, and filtered after standing two hours. The supernatant liquid is decanted through a filter-paper, and the precipitate washed by decantation several times with a 2 per cent. sodium nitrate solution to prevent the deflocculation of the precipitate. The filter is then washed into the beaker, and the precipitate dissolved in standard alkali and titrated back, using phenolphthaleïn as indicator. The presence of excess of alkali results in the production of free ammonia in the liquid, necessitating rapid titration and the use of at least 12 drops of the indicator made up in the usual way. The factor recommended for $\frac{N}{10}$ alkali is 1 c.c. = 0·0003004 P_2O_5. H. F. E. H.

New Physico-Chemical Volumetric Method. R. Dubrisay. (*Bull. Soc. Chem.*, 1914, **15**, 444-451.)—The method described previously (ANALYST, 1913, **38**, 294) may be used for the titration of phenol and of chromic and periodic acids with satisfactory results. It may be employed for the titrations of very dilute solutions, but is not applicable in the case of concentrated solutions containing hydrolysable salts. W. P. S.

Estimation of Potassium as Potassium Hydrogen Tartrate. F. Marshall. (*Chem. Zeit.*, 1914, **38**, 585-587, 615-616.)—In the absence of other metals, of phosphoric acid and silica, and more than traces of ammonium salts, 0·05 grm. potassium may be separated from an equal weight of sodium as follows: The solution of the alkalis as chlorides is evaporated to dryness, the residue dissolved in the smallest possible quantity of water (3 to 4 drops), and the potassium precipitated by the addition of 20 c.c. of a 2 per cent. alcoholic solution of tartaric acid saturated with potassium hydrogen tartrate. From 10 to 20 c.c. of 96 per cent. alcohol, also saturated with potassium hydrogen tartrate, is added, the mixture boiled for ten minutes on the water-bath, and then allowed to stand for twenty-four hours (not less), with frequent shaking. The precipitate of potassium hydrogen tartrate is finally filtered off on a Gooch crucible, washed with alcohol saturated with potassium hydrogen tartrate, dried at 80° C., and weighed. The solubility of potassium hydrogen tartrate in 96 per cent. alcohol is only 3 mgrms. per 100 c.c., and is independent of the temperature between 15° C. and the boiling-point of the liquid. With the quantities named, the maximum error of a single estimation is $-1·4$ mgrms., the average error $-0·5$ mgrm. No experiments with larger quantities are described. G. C. J.

Errors in the Estimation of Rare Earths as Hydroxides. T. O. Smith and C. James. (*J. Amer. Chem. Soc.*, 1914, **36**, 909-912.)—It has been observed that when the rare earths are precipitated as hydroxides and ignited to oxides, the results are always somewhat higher, and sometimes much higher, than when they are precipitated as oxalates and ignited. Moreover, the former method gives less concordant results. This latter circumstance suggested the possible formation of basic salts, the amount of which might be expected to vary with the experimental conditions. The point was investigated, using lanthanum chloride, lanthana being the most powerful base of the rare earths, and therefore most likely to form basic salts, and chlorine being more likely than nitric acid to be held by the lanthanum hydroxide on ignition. At the same time the possibility of the oxalate precipitation being incomplete in presence of a trace of mineral acid was also investigated.

It was found that oxalic acid precipitated lanthana quantitatively from solutions very faintly acid with hydrochloric acid. The complete absence of free mineral acid can be insured by using excess of ammonium oxalate instead of oxalic acid as precipitant, but the method is likely to introduce errors, as the precipitate carries down ammonium oxalate when this is present in excess; and when the complex ammonium lanthanum oxalate is washed, it appears to undergo hydrolysis with the production of lanthanum oxalate so finely divided that it passes through the filter. The use of sodium hydroxide undoubtedly gives rise to results above the truth, and these are due mainly to the retention of sodium by the precipitate, and not to the formation of basic salt, since the results are still higher when a large excess of sodium hydroxide is used. In the case of lanthanum chloride, precipitation with ammonia also gives rise to high results, which in this case are due to the formation of basic chloride. The error in this case is 0·7 per cent., and the opinion is expressed that, with the nitrate, the error would be negligible. With sodium hydroxide, the error cannot be kept below 1·4 per cent., and may be very much greater if a large excess of the reagent is used. G. C. J.

Volumetric Estimation of Sulphurous Acid. E. Kedesdy. (*Chem. Zeit.*, 1914, **38**, 601.)—Phenolphthaleïn is not suitable for the estimation of sulphurous acid, as it does not give a sharp end-point. For the estimation of sulphur dioxide in *oleum* the following method is recommended: The total acidity is first determined with standard sodium hydroxide and methyl orange; the sulphurous acid or sulphite is then converted into sulphate and free sulphuric acid by excess of hydrogen peroxide, and the liquid again titrated; the alkali now used is equivalent to half the sulphurous acid originally present. O. E. M.

Use of Telluric Acid in Estimation of Bromine associated with Chlorine in Haloid Salts. F. A. Gooch and H. I. Cole. (*Zeitsch. anorg. Chem.*, 1914, **86**, 401-406).—Gooch and Blumenthal have described how selenic acid may be applied to this purpose (ANALYST, 1913, **38**, 227). Telluric acid is not strong enough to set free hydrogen bromide from metallic bromides, and a stronger acid must therefore be present before use can be made of the capacity of telluric acid to set free bromine from hydrogen bromide. In presence of a suitable quantity of sulphuric acid, however, bromine may be quantitatively expelled from a mixture of chloride and bromide without any evolution of chlorine, provided certain conditions as to concentration are complied with. The mixed haloid salts, or their solution, are introduced together with about 1 grm. of telluric acid into a Voit's flask, the side tube of which has sealed to it a stoppered tap funnel, whilst the neck is closed by a ground stopper carrying a right-angled bend. This bend is sealed to the inlet bend of a Drechsel's wash-bottle charged with 300 c.c. of 1 per cent. potassium iodide solution and cooled in ice. To the outlet of the Drechsel bottle is sealed a bulb absorption apparatus, also charged with potassium iodide solution. The volume of the solution of haloid salt having been brought up to 40 c.c., 10 c.c. of dilute (1 : 1) sulphuric acid is added to the contents of the Voit flask, the tubulure of the tap funnel is connected to a Kipp's apparatus generating carbon dioxide, and a current of that gas passed slowly through the whole train of apparatus. The contents of the Voit flask are meanwhile heated, slowly at first, but finally to boiling, and the boiling continued until all but about 17 to 18 c.c. has distilled. The iodine liberated by the bromine is then titrated in the usual manner.

If the concentration of the acid mixture in the Voit flask be pushed too far, chlorine will be evolved; whilst if not carried far enough, the expulsion of bromine will be incomplete. With a final concentration of 20 c.c. the error is negligible, if chlorides are absent, but in presence of much chloride, bromine may be underestimated by 0·4 per cent. With a final concentration of 15 c.c., so much chlorine may be set free as to cause the bromine to be overestimated by 0·4 per cent. With a final concentration of 10 c.c. the error may be no greater if the bromine-chlorine ratio is high, but small quantities of bromine in presence of much chloride may be seriously overestimated. Under the conditions laid down the results are excellent, and to realise these conditions it is suggested that the Voit flask should be roughly graduated. G. C. J.

Use of Ammonium Salt of Nitrosophenylhydroxylamine (Cupferron) in Quantitative Separation of Titanium from Iron. W. M. Thornton, jun. (*Zeitsch. anorg. Chem.*, 1914, **86**, 407-412.)—Gooch's method for the separation of iron and titanium, by the precipitation of the former as sulphide in presence of tartaric acid, is attended with inconvenience, as the tartaric acid in the filtrate has to be destroyed before the titanium can be precipitated by the usual reagents. Destruction of the tartaric acid by permanganate, as Gooch directs, generally leads to co-precipitation of manganese when the attempt is made to precipitate the titania by hydrolysis of the acetate, and a second separation is thus necessitated. The author has found that Baudisch's (ANALYST, 1911, **36**, 520) reagent, "cupferron," will precipitate titanium from solutions containing large quantities of tartaric acid. The following method of separating iron from titanium is based on this observation:

To the ferric solution, tartaric acid in amount not less than three times the weight of the ferric oxide and titania is added. The solution is neutralised with ammonia if strongly acid, and then reacidified by addition of 1 to 2 c.c. of dilute (1 : 1) sulphuric acid and diluted to 100 c.c. Hydrogen sulphide is led into the solution until it appears colourless. It is important to effect this reduction in acid solution, otherwise the ferrous sulphide may carry down some titania. The reduced solution is next made distinctly alkaline with ammonia, and hydrogen sulphide is again passed until all the iron is precipitated. The precipitate is filtered off and washed with very dilute colourless ammonium sulphide. The filtrate is acidified with 12 c.c. of dilute (1 : 1) sulphuric acid, boiled to expel hydrogen sulphide, partly neutralised so that it contains about 2·5 grms. of real sulphuric acid in each 100 c.c., and allowed to cool. A sufficient quantity of a 6 per cent. solution of Baudisch's reagent is added, and the precipitate given time to subside. Addition of more of the reagent should give rise only to a white precipitate of nitrosophenylhydroxylamine, a yellow precipitate indicating that too little was used in the first instance. The precipitate is filtered off on an ordinary filter, washed with cold water, dried in a crucible, decomposed by cautious heating, and finally ignited. A Gooch crucible is not suitable for the filtration, as the precipitate appears to fuse when dried at 110° C. For the same reason the filter should not be dried in its funnel, but in a crucible, as directed. The maximum error of the method is ±0·4 mgrm., with quantities of titanium dioxide up to 0·15 grm. G. C. J.

Colorimetric Estimation of Iron in Water. F. Gothe. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1914, **27**, 676-683.)—The thiocyanate colorimetric method of estimating iron in water tends to yield untrustworthy results when the iron has been previously oxidised by treatment with hydrochloric acid and potassium chlorate; this is due to the interfering action of the excess of the acid on the coloration obtained. The author shows that the maximum coloration is obtained when the concentration of the hydrochloric acid in the test solution is kept within very narrow limits, and gives the following procedure for obtaining reliable results with the method. One hundred c.c. of the water are evaporated to dryness after the addition of 1 c.c. of hydrochloric acid (sp. gr. 1·125) and a few crystals of potassium chlorate; 1 c.c. of the hydrochloric acid is added to the residue, then water, and the solution is diluted to 95 c.c. in a graduated cylinder. After the addition of 5 c.c. of a 10 per cent. potassium thiocyanate solution, the coloration obtained is compared with that produced by a known quantity of iron under similar conditions. In the case of waters containing large quantities of organic matters, these must be destroyed by ignition before the iron is estimated.

The addition of a considerable excess of thiocyanate is essential to obtain the maximum coloration. Although nitric acid, when used as the oxidant for the ferrous salts, does not affect the intensity of the coloration to the same extent as does hydrochloric acid, variations in the colour may be noticed with relatively widely varying concentrations of the acid. W. P. S.

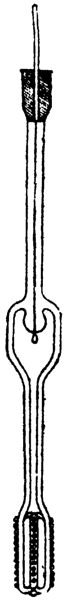
APPARATUS, ETC.

Apparatus for Catalytic Reductions with Hydrogen. H. Voswinckel. (*Chem. Zeit.*, 1914, **38**, 634.)—This is a portable apparatus, comprising two graduated pressure flasks, communicating by means of tubulures at the bottom, and terminating at the top in tubes bent twice at right angles. These tubes are connected with the hydrogen tube, and the gas passing through a system of taps enters alternately into each of the flasks. The flasks are charged with about 1½ litres of water, which, when one flask is filled with hydrogen, passes into the other flask. After the hydrogen has been brought to the desired pressure by means of a reducing valve, the reaction vessel is connected with the tube of the flask and shaken in a machine. If all the hydrogen in this flask has been absorbed, the other flask is filled with gas and the reduction continued without interruption. In this way a series of reductions may be made with a definite volume of hydrogen at a definite pressure.

C. A. M.

Apparatus for Testing the Volatility or Oxidisability of Oils. H. Kantorowicz. (*Zeitsch. angew. Chem.*, 1914, **27**, 272.)—The dish containing the weighed quantity of the oil is placed in an inner vessel, through which is passed a current of air or gas. This vessel is surrounded by a bath of naphthalene or other suitable substance, which is heated by a Bunsen burner. After the exposure the oil is weighed, and the tar and rosin values and increase of asphaltum estimated. The apparatus may be obtained from Muencke, Berlin. C. A. M.

Rotating Cathode and Anode for Rapid Electrolytic Analysis. E. A. Lewis. (*J. Soc. Chem. Ind.*, 1914, **33**, 445.)—The cathode, used in zinc or copper estimations, is made of 20-mesh copper gauze, 7·5 cm. by 4 cm., supported by stiff copper wire; for use in acetic acid or ammoniacal solutions it must be made of silver, or silver plated. The anode consists of 14 inches of platinum wire weighing one grain to the inch, wound upon a frame of glass rod, and connected by means of bell wire. O. E. M.



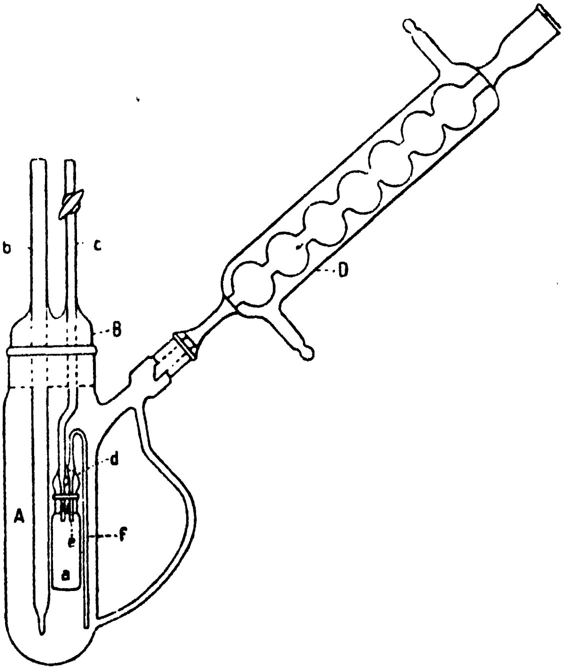
Apparatus for Estimation of Small Quantities by Electrolysis with the Rotating Cathode. R. Heinze. (*Zeitsch. angew. Chem.*, 1914, **27**, 237-240.)—The cathode consists of a length of fine platinum or gold wire, wound in the form of a helix round the lower end of the cathode holder illustrated, the glass points serving to keep the several turns of the helix apart. Wire of 0·1 mm. diameter serves well; its length must depend on the load which the micro-balance will accommodate; but 15 cm. of gold wire or 13·5 cm. of platinum wire of the diameter stated can be conveniently weighed on a Nernst balance as sent out by Spindler and Hoyer, Göttingen. The glass holder is about 10-11 cm. long over all, the upper straight tube being about 4 mm. in diameter. This tube carries a 0·3 mm. platinum wire, which pierces its lower end, and is there bent into a loop, whilst the upper end of the platinum wire is soldered to a stout (2 mm.) copper wire, the joint being embedded in Canada balsam. From the main stem of the holder two 2 mm. tubes spring out, then approach each other, run parallel for some distance, and are finally sealed to the bundle of four small glass rods, about 15 mm. long, round which the cathode wire is wound. One end of the cathode wire is, of course, twisted round the platinum loop, which connects through the copper lead to the source of electrical energy.

The anode consists of about 25 cm. of 0·25 mm. platinum wire, and the paper includes an illustration of a simple glass frame on which such an anode may conveniently be mounted. The paper also includes illustrations of all the subsidiary apparatus, which does not differ from that in use when working with larger quantities of material. The manipulation of the wire cathode, so as to transfer it to the balance without touching it with the hands, is also described.

Test numbers are given which show that the apparatus will serve for the estimation of 0·2 mgrm. of mercury in presence of 20 mgrms. of zinc, with an error not exceeding 1 per cent., and it is said that the apparatus makes it possible to apply all rapid methods of electro-analysis to the estimation of quantities of this order of magnitude. Details as to current density, time required for complete deposition, composition of electrolyte, etc., will be given in another paper. G. C. J.

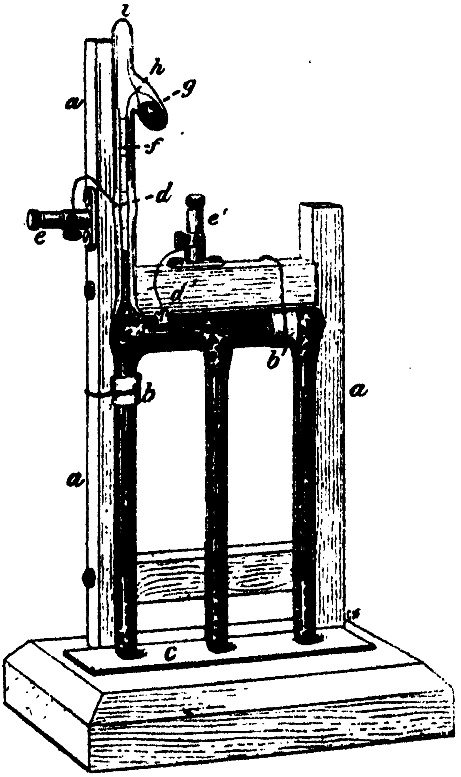
New Method for Determining Solubilities at High Temperatures.

L. Tschugaeff and W. Chlopin. (*Zeitsch. anorg. Chem.*, 1914, **86**, 154-162.)—The solubility, and especially the curve expressing change of solubility with change of temperature, are valuable criteria in deciding the identity of a substance, and also in many cases in deciding whether it is accompanied by impurities, many of which have a marked influence on the solubility. Whereas other physical constants are invariably determined for new substances, concerning many there are no solubility data, or at most a single value for some one temperature. The authors attribute this to the difficulty attending solubility determinations by the methods generally in use, usually modifications of Noyes' or Victor Meyer's method. When using Noyes' method, great difficulty is experienced in getting accurate results at high temperatures, and though V. Meyer's method gives results of sufficient accuracy, it requires the use of a very large thermostat which must be maintained constant within 0·1° C.



The principle of the method now described is the determination of solubilities at the boiling-point of the saturated solution, the boiling-point being varied by changing the pressure. Conducting the determination at the boiling-point ensures thorough agitation, and eliminates the necessity for using a thermostat, since at constant pressure the boiling-point is also constant.

The apparatus figured consists of the large vessel *A*, into which the substance is introduced, together with enough of the solvent to fill it to some point between *a* and *e*. The stopper *B* carries two tubes, one of which, *b*, accommodates a thermometer divided into tenths of fifths of a degree, the bulb of the thermometer being wholly in the liquid. The tube *c* carries at its lower end the stopper of the weighing bottle *a*, which is wired to the stopper, glass points being attached to each to facilitate this. The other tube, *d*, passing through the stopper, and ending in the syphon-tube, *f*, has an enlargement which serves to accommodate asbestos, cotton-wool, or other suitable filtering material. In use, the upper end of the condenser is connected with a water-pump, interposing a mercury pressure regulator or for less exact work two large empty vessels, and the tubes *b* and *c* have fitted to them pieces of rubber tube carrying pinch-cocks. By regulating these pinch-cocks the pressure is adjusted to any desired value, and the apparatus is placed in an oil-bath 5 to 10 degrees above the boiling-point of the solution for an hour. If the vacuum is sufficiently high, the weighing bottle *a* may be filled by closing the cock on *c* and breaking the vacuum; if the vacuum is not sufficiently high for this, *c* is connected to the pump for a few seconds. The weighing bottle *a* is now removed, wiped, and weighed, and its contents are evaporated and the residue weighed, or the concentration determined in any other convenient manner. G. C. J.



Thermo-Regulator for Electrically Regulated Constant Temperature Chambers. H. H. Bunzel and H. Hasselbring. (*J. Amer. Chem. Soc.*, 1914, **36**, 949-951.)—The form of the mercury container is not material. That illustrated—a horizontal cylinder of 1 cm. diameter, with three vertical tubes each 10 cm. long—has been found convenient where the regulator is placed directly in the chamber to be controlled. Where the temperature is indirectly controlled, as in a water-jacketed incubator, an oblong bulb would be more convenient. The novel principle involved is the provision of the auxiliary bulb, *g*, connected with the stem of the regulator by a capillary neck, *h*. This bulb, the contents of which are about one-fortieth of that of the main mercury container, serves, like the top bulb of a Beckmann thermometer, to make the instrument adaptable to a wide range of temperatures. Before sealing off at *i*, the mercury in the apparatus is boiled under reduced pressure to expel air, and thus avoid corrosion of the mercury surfaces, due to sparking at the point of contact with the wire *d*. In order to avoid sparking, the regulator should be used with a very small current (0·01 ampère or less), which actuates a relay. The heating current cannot be passed through the apparatus. The transference of the right quantity of mercury from the auxiliary bulb to the regulator, or conversely, is made easier by engraving a short scale on the stem for 1 or 2 cm. above and below *d*. G. C. J.

AUGUST, 1914. Vol. XXXIX., No. 461.

THE ANALYST.

ANALYTICAL CHEMISTRY INVESTIGATION SCHEME.

THE Honorary Secretaries of the Society of Public Analysts and other Analytical Chemists have been instructed to draw attention to the scheme for promoting research in Analytical Chemistry, which was initiated by the present President and the Council of the Society about ten years ago. A statement showing the circumstances which led to its adoption was published in the ANALYST of February, 1908, and it has been thought desirable at the present time to restate its objects and to record the results obtained.

Members of the Society when reading abstracts of papers appearing in British and foreign journals, frequently meet with descriptions of new analytical processes which would appear to be of exceptional utility and value. Since these emanate very often from private laboratories, and are the work of men whose names are practically unknown outside a limited circle, the results stand in need of confirmation before they can be generally accepted.

Originally it was intended to restrict the revision of such pieces of work to members of the Society, and although this was done in some cases, it was felt on further consideration that the scheme should be extended both as regards the workers and subjects dealt with.

Analysts with large practices, and more particularly those who specialise, are frequently brought face to face with analytical problems, the accurate study and careful solution of which would be of the very greatest value, and it was felt that the idea above referred to might well be extended to cover original investigations of this character. The following, therefore, is the kind of work with which the scheme proposed to deal:

1. The investigation of analytical processes, and of problems in analytical chemistry.

2. The revision of certain published processes, and, assuming it to be desirable, their extended study.

The Council having decided to enlist the co-operation of workers outside the membership of the Society, it was suggested that such investigations might be undertaken by some of the senior students working in the laboratories of the larger colleges and teaching institutions. In such laboratories there are frequently students who are being trained for the profession of analytical chemistry, and who have arrived at a point in their studies when original problems are put into their hands by their professors. It was thought that if important subjects for investigation in the domain of analytical chemistry were suggested to the professor by some competent specialist or body of specialists, he might be willing to allot them to some of his senior students, and to maintain a careful supervision over the execution of the work. It was pointed out that this scheme would not interfere in any way with the research work being done in other branches of the science, and that students in course of training for a professorial career, or those specialising in certain departments of chemistry, would continue to concern themselves with such researches as were suggested to them by their professors, the analytical investigations being assigned to students working with the intention of becoming analytical or technical chemists. It seemed probable that these would take special interest in such investigations, whilst there could be no doubt that their work, if carefully done, would be of great value. It had to be remembered, moreover, that in the case of candidates for the examinations of the Institute of Chemistry, such investigations would be taken into account by the examiners in deciding the examinations.

That these expectations have been fully realised is shown by the results obtained under the scheme. Since its initiation the following thirty-six investigations, many of which constitute very useful additions to the science and practice of analytical chemistry, have been completed and published in the ANALYST, whilst a number of investigations are now in hand.

The following is a list of papers published under the scheme :

Julian L. Baker and W. D. Dick : "Detection and Estimation of Small Quantities of Maltose in the Presence of Dextrose."

D. L. Howard : "Note on the Separation of Strychnine and Brucine."

P. Schidrowitz and F. Kaye : "The Determination of Higher Alcohols in Spirits" (No. 1).

P. Schidrowitz and F. Kaye : "The Determination of Higher Alcohols in Spirits" (No. 2).

S. W. Collins : "The 'Nitron' Method for the Estimation of Nitric Acid."

L. Gowing-Scopes : "The Estimation of Tartaric Acid in the Presence of Malic and Succinic Acids."

L. E. Hinkel : "The Detection of Small Quantities of Methyl Alcohol in the Presence of Ethyl Alcohol."

Z. Kahan : "Quantitative Separation of Barium from Strontium."

F. M. Perkin and T. W. Harrison : "The Action of Dimethyl Sulphate upon Oils of the Aromatic and Aliphatic Series."

E. Cahen : "The Separation of Antimony and Tin."

G. G. Henderson and J. C. Galletly : "The Quantitative Separation of Lead and Bismuth."

P. J. Narracott : "Experiments on the Separation of Mixtures of some Aliphatic Acids by Means of Benzene."

G. B. Neave : "The Sabatier-Senderens Test for Distinguishing between Primary, Secondary, and Tertiary Alcohols."

G. B. Neave : "Note on a Proposed Test for Halogens."

E. Cahen : "Comparison of Pozzi-Escot's and Devarda's Method for the Estimation of Nitrates."

E. Cahen and H. F. V. Little: "The Separation of Bismuth from Lead, and the Analysis of Bismuth-Lead Alloys."

A. Gemmell: "Improved Method for the Estimation of Titanium."

L. Gowing-Scopes: "The Uses of Trichlorethylene in Analytical Chemistry."

E. L. Rhead: "Report on Gravimetric Methods for the Determination of Nickel in Nickel Steel."

E. Cahen and H. F. V. Little: "Waldemar Fischer's Modification of Volhard's Method for the Volumetric Estimation of Manganese, and its Comparison with other Well-Known Methods."

T. Cockburn and J. W. Black: "The Estimation of Quinine as the Acid Citrate in Certain Organic Liquids."

W. L. Austin and Charles A. Keane: "The Analysis of Lithopone."

C. O. Bannister and W. McNamara: "The Effect of Lime on the Ammonium Molybdate Lead Assay."

S. W. Collins: "The Separation of Arsenic from Antimony and other Metals, with some Application to Toxicological Work."

L. Eynon and J. Henry Lane: "The Estimation of Furfural by Means of Fehling's Solution."

N. Evers: "The Detection and Estimation of Arachis Oil."

G. B. Neave: "Solubilities of the Lead Salts of the Higher Fatty Acids in Ether and in Petroleum Ether."

G. D. Elsdon: "Alcoholysis and the Composition of Coconut Oil."

L. Gowing-Scopes: "The Estimation of Citric Acid in the Presence of Certain Other Acids."

G. D. Lander and H. W. Winter: "Antipyrin in Toxicological Analysis."

H. L. Smith: "The Determination of Tannin in Tea."

C. O. Bannister and E. A. du Vergier: "The Determination of Iridium in Platinum-Iridium Alloys."

G. D. Elsdon: "The Composition of Palm-Kernel Oil."

A. Gemmell: "The Insoluble Bromide Value of Oils and its Determination."

L. Gowing-Scopes: "The Properties of some Chlorhydrocarbons and their Uses in Chemical Analysis" (Part 1).

L. Gowing-Scopes: "The Properties of some Chlorhydrocarbons and their Uses in Chemical Analysis" (Part 2).

The following are the only conditions in connection with this scheme:

1. The matters for investigation to be in all cases subject to the approval of the Publication Committee of the Society.

2. An account of the work when completed to be published, subject to the approval of the Publication Committee, in the ANALYST, and, when possible, to be brought before the Society in the form of a communication.

A fund has been established, and investigators undertaking work under this scheme are entitled to make a claim on this for any special apparatus or chemicals they may require. In addition, members of the Society having special knowledge of any of the subjects being dealt with are always very willing to assist investigators either with their advice or by supplying them with specially pure materials or specimens, should they happen to possess such. All papers are published under the names of the investigators, and due recognition is given to the college or institution in which the work has been done.

The Council are earnestly desirous of enlisting the sympathy and active co-operation of all members of the Society and others interested in analytical chemistry.

The scheme may be supported in three ways—viz.:

1. By suggesting suitable subjects for investigation.
2. By undertaking investigations so suggested, or by inducing students or assistants to undertake such work.
3. By contributing to the Analytical Chemistry Investigation Scheme fund.

Those who are willing to support this scheme in one or more of these directions are requested to communicate with the honorary secretaries.

* * * * *

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

THE DETERMINATION OF IRIDIUM IN PLATINUM-IRIDIUM ALLOYS.

By C. O. BANNISTER, A.R.S.M., F.I.C., and E. A. DU VERGIER.

Carried out under the Analytical Investigation Scheme of the Society of Public Analysts and other Analytical Chemists.

(Read at the Meeting, June 3, 1914.)

Owing to the increasing value of platinum-iridium alloys and to the large number of industrial purposes to which these alloys are now being applied, the accurate determination of iridium is of considerable importance. The following notes are put forward in order to indicate the best methods of detecting the presence of and determining the amount of iridium in these alloys. As platinum-iridium wires are largely used in conjunction with wires of pure platinum in thermo-electric pyrometry, it is often desirable to be able to differentiate between these two materials, and as platinum-rhodium wires are frequently used in the place of the platinum-iridium wires, it is also desirable to be able to readily decide whether iridium or rhodium is the alloying metal. In a recent communication to the Institution of Mining and Metallurgy (Bannister and Patchin, "A Simple Method for the Detection of the Platinum Metals in Cupellation Beads." Bulletin No. 111, December, 1913), the differentiation between platinum-iridium and platinum-rhodium alloys was shown to be quite a simple matter. In this case advantage is taken of the fact that in the presence of small quantities of rhodium the tendency of silver cupellation beads to spit or vegetate during solidification is greatly increased.

In order to make use of this phenomenon as a means of identification of rhodium in alloy with platinum, from 10 to 20 mgrms. of the alloy are cupelled with about 2 grms. of silver and 12 to 15 grms. of lead, and if the resulting beads show excessive and uncontrollable spitting, the presence of rhodium may be inferred. Small quantities of rhodium also cause distinct surface crystallisation on the silver beads, the facets of the crystals being visible to the naked eye. When the amount of rhodium present amounts to 0·3 per cent. or more the silver beads assume a bluish-grey colour, whilst the metal extruded during the spitting is silver-white in colour. As neither platinum nor iridium give these same indications when cupelled with silver, it will be seen that a simple cupellation operation may be used to determine the presence of rhodium in platinum or platinum alloys.

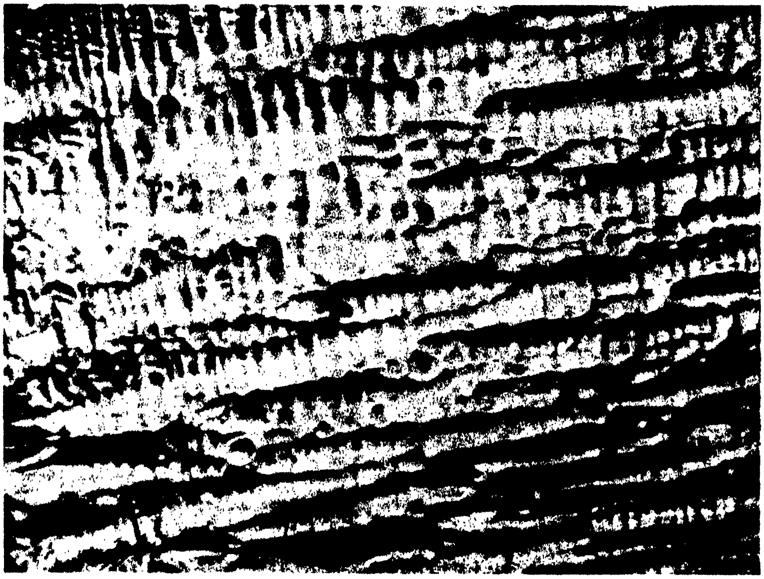


FIG. 1.—SILVER CONTAINING 0·8 PER CENT. OF PLATINUM.

In the paper already mentioned, it was also shown that silver cupellation beads containing between 0·3 and 1·6 per cent. of platinum, when examined under a low power microscope showed a peculiar corrugated structure, as illustrated in Fig. 1. This structure is quite characteristic and very different from that obtained with silver containing no platinum (Fig. 2), but is similar to that obtained when palladium is present. In a similar manner, it was found possible to detect iridium in silver beads, for this metal caused the beads to assume a more spherical form than that usual with pure silver, the surface at the same time being covered with lines crossing one another after the manner of slip bands (Fig. 3). The shape of these beads and the surface markings have been found to be associated with internal blowholes, and there is little doubt that they are caused by the liberation of oxygen after a certain thickness of silver has solidified and the surface has become sufficiently strong to resist fracture. The beads thus become stressed during the liberation of the oxygen, and the result is seen in the stress marks already mentioned.

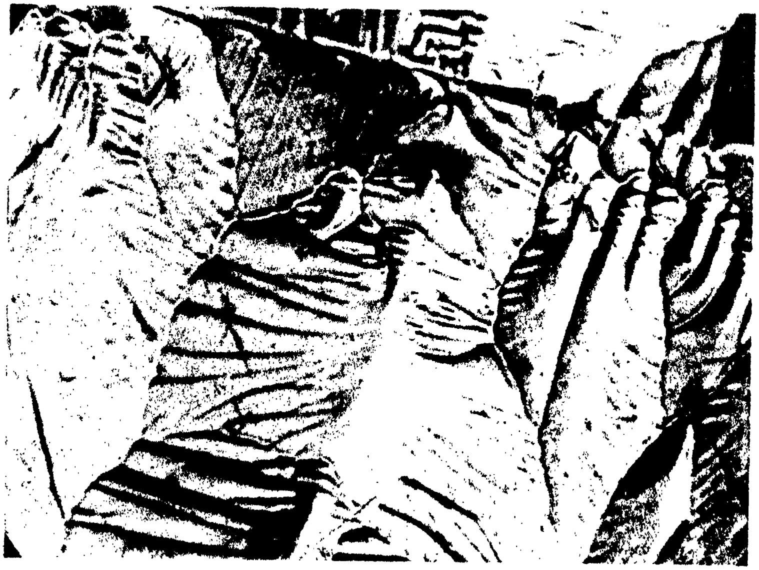


Fig. 2.—Silver.

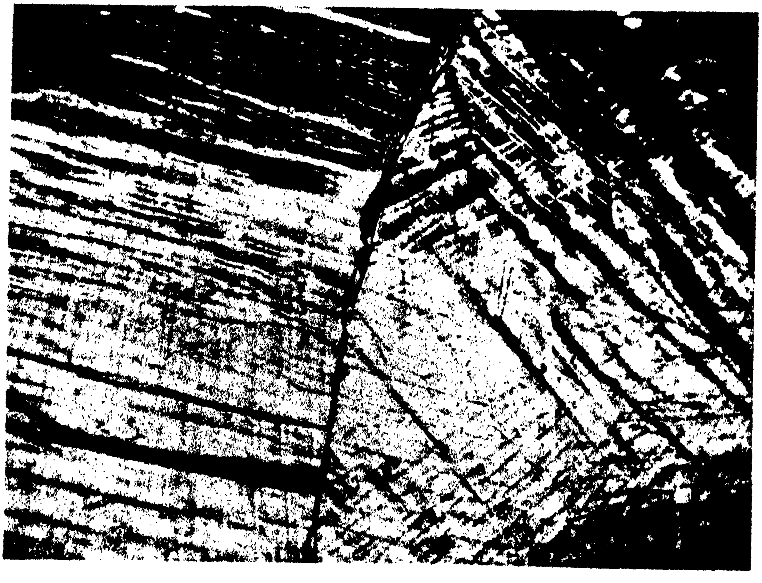


Fig. 3.—Silver containing 1·8 per Cent. of Iridium.

Unfortunately, the presence of platinum is found to mask this characteristic effect of iridium, and for its detection in platinum-iridium alloys it is necessary, after a first cupellation of 0·05 grm. of the alloy with 2·0 grms. of silver, to part the bead in sulphuric acid, to separate most of the platinum by means of *aqua regia*, and to recupel the residue with a further quantity of 2 grms. silver. After these two operations have been carried out, distinct indications will be found on examining the beads under a low-power objective, if iridium be present.

In the actual determination of iridium in platinum-iridium alloys, it is not possible to separate and determine the iridium by simply digesting in *aqua regia*, as the relative solubility of platinum and iridium varies with the composition of the alloys. This method of separation cannot be used when the two metals are alloyed, and can only be applied after a preliminary operation has been carried out to separate the platinum and iridium into distinct particles.

In order to ascertain the degree of solubility of platinum and iridium alloyed together in *aqua regia*, a series of alloys containing 2·5, 5·0, 10·0, 20·0, and 25·0 per cent. of iridium was examined as follows: The alloy wires were first annealed at a high temperature, then rolled as thin as possible, being annealed several times during the rolling operation. A portion of each alloy was then digested in dilute *aqua regia* of varying strength, but only small quantities of platinum and iridium passed into solution. With fairly strong *aqua regia*, however, the action was found to be considerable, in some cases the whole of the alloy passing into solution. An accurately weighed portion—approximately 0·5 grm. of each of the alloys—was digested on a hot plate with 10 c.c. of *aqua regia*, made up in the proportions HCl (4 vols.), HNO_3 (1 vol.), H_2O (1 vol.). When evaporated nearly to dryness, a further 10 c.c. of the *aqua regia* was added, again digested, and so on, until no further action was observed. The acid was now evaporated down to 5 c.c., diluted, and decanted. The insoluble residue, which retained the original form of the flattened wire, was washed by decantation, dried, annealed, and weighed. The following table gives details of this experiment:

Alloy.	Weight taken.	*Aqua Regia.*	Time.	Insoluble Residue.	Soluble.
Per Cent.	Grm.	C.C.			Per Cent.
1. Iridium 2·5	0·5418	20	Entirely soluble in 2 hours	Nil	100
2. Iridium 5·0	0·5488	25	Entirely soluble in 2½ hours	Nil	100
3. Iridium 10·0	0·5422	30	Digested for 4¼ hours	0·0505 = 9·32 per cent.	90·68
4. Iridium 20·0	0·5508	40	Digested for 4¼ hours	0·5400 = 98·04 per cent.	1·96
5. Iridium 25·0	0·5534	40	Digested for 4¼ hours	0·5492 = 99·24 per cent.	0·76

The solutions resulting from the treatment of alloys 3, 4, and 5 with *aqua regia* were examined for platinum and iridium by evaporating nearly to dryness, to expel nitric acid, followed by further evaporation after the addition of hydrochloric acid. The platinum and iridium chlorides thus obtained were dissolved in a small quantity of water, and the platinum and iridium precipitated with a saturated solution of ammonium chloride in the presence of alcohol. The variation of the colour of the precipitate from lemon yellow to reddish-brown is a useful indication as to the amount of iridium present.

The mixed precipitate of ammonium platinichloride and ammonium iridiochloride was washed with ammonium chloride and alcohol. It was found necessary to treat the filtrate from this precipitate for traces of platinum and iridium, and this was carried out by the addition of a few drops of hydrochloric acid and a small quantity of pure granulated zinc. This was allowed to stand until all the platinum and iridium was precipitated and the zinc dissolved. The residue was washed with hot water and then dilute nitric acid, followed by hot water, and added to the mixed ammonium chloroplatinate and chloroiridiate precipitate. This mixture was now ignited, first at a low temperature to drive off ammonium chloride, then at a high temperature for some time to reduce the platinum and iridium to the metallic state. The platinum-iridium sponge thus obtained was digested with dilute *aqua regia* (HCl 4 vols., HNO 1 vol., H_2O 10 vols.) until the platinum dissolved, and the black insoluble residue of iridium was washed, dried, ignited at a high temperature, and weighed. The platinum in solution was precipitated, as already described, and the metallic sponge heated in a current of hydrogen and weighed. The following figures show the amount of platinum and iridium passing into solution :

Alloy.	Amount Soluble.	Platinum in Soluble Portion.	Iridium in Soluble Portion.
Per Cent.	Per Cent.	Per Cent.	Per Cent.
3. Iridium 10·0	90·68	96·0	4·0
4. Iridium 20·0	1·96	89·0	11·0
5. Iridium 25·0	0·76	83·3	16·7

The method given above is not recommended as a method of analysis, even in cases where it is possible to get the whole of the alloy in solution in *aqua regia.* The spongy mixture of platinum and iridium obtained by the ignition of the ammonium platinichloride and iridiochloride contains the iridium in such an extremely fine state of subdivision that a portion of the iridium is attacked even by dilute *aqua regia*, and the results for iridium are likely to be low.

For the accurate determination of iridium in these alloys, it is best to melt with silver or lead, during which operation the platinum alloys with the added metal and the iridium present in the original alloy is liberated in the free state, and may be separated. For a comparison of these two methods, alloys were examined containing 20 and 25 per cent. iridium.

Method 1.—Alloying with Silver and Parting.

In this method a parting alloy was first prepared by cupelling 0·25 grm. of the alloy with 1·5 grms. pure silver and 10 grms. assay lead. The cupellation was finished at a high temperature to expel the last traces of lead. The resulting button was cleaned, hammered, annealed, and rolled into the usual cornet form. This was parted in assay flasks with 40 c.c. concentrated sulphuric acid. After the first treatment with acid, the silver sulphate solution was decanted and the residue again boiled with 40 c.c. concentrated sulphuric acid. The solution was decanted, and the residue washed twice with water, and then transferred to an annealing cup. The platinum-iridium residue was dried and annealed at a high temperature and weighed.

The following are the weights obtained by experiment:

	20 per Cent. Iridium Alloy.	25 per Cent. Iridium Alloy.
Experiment 1	0·2512 grm.	0·2514 grm.
„ 2	0·2508 „	0·2510 „

The increase in weight thus obtained is due to the presence of small quantities of silver. The platinum-iridium residue thus obtained was digested with 10 c.c. dilute *aqua regia* (HCl 4 vols., HNO_3 1 vol., H_2O 10 vols.), until action ceased, then evaporated to about half its original volume. The solution was decanted and the residue again digested with 5 c.c. dilute *aqua regia* as above. The two solutions thus obtained were now mixed, diluted to 25 c.c., boiled, and filtered. The residue thus obtained consists of iridium, together with some silver chloride, and was washed with dilute ammonia to dissolve this silver chloride, then washed with hot water, dried, ignited at a high temperature, and weighed. The following are the results obtained:

	20 per Cent. Iridium Alloy.	25 per Cent. Iridium Alloy.
Experiment 1 ...	0·0505 grm. iridium = 20·20%	0·0630 grm. iridium = 25·20%
„ 2 ...	0·0510 „ = 20·40%	0·0631 „ = 25·24%

The filtrate obtained by the treatment of the platinum-iridium residue with *aqua regia* was evaporated nearly to dryness to expel nitric acid, a few drops of hydrochloric acid added, and the solution diluted to 50 c.c. The platinum was now precipitated with pure zinc (free from lead, etc.), allowed to stand until the zinc was dissolved and the platinum precipitated, then the platinum was filtered off, washed with dilute nitric acid and hot water, dried, ignited at a high temperature, finally heated for ten minutes in an atmosphere of hydrogen, cooled, and weighed. The following are the results obtained:

	20 per Cent. Iridium Alloy.	25 per Cent. Iridium Alloy.
Experiment 1...	0·1994 grm. platinum = 79·76%	0·1867 grm. platinum = 74·68%
„ 2...	0·1988 „ = 79·52%	0·1864 „ = 74·56%

Method 2.—Alloying with Lead and Parting.

This method was suggested in a paper by the late George Matthey, on the "Preparation of Pure Platinum and Iridium" (Proc. Royal Soc., 1879, 28, 463).

Half a grm. of the alloy was melted down with 5 grms. pure assay lead in a carbon crucible. The carbon crucible containing the lead and alloy was packed in a Cornish crucible with small pieces of charcoal, covered with a lid, and heated to a high temperature in a wind furnace for one hour. The lead was allowed to cool slowly in the crucible. By this means the heavy iridium settles to the bottom of the lead. The lead button obtained was digested with 50 c.c. dilute nitric acid (1 to 10). When action ceased, the insoluble residue was allowed to settle and the solution decanted whilst hot. The residue was again digested with 50 c.c. dilute nitric acid as before. The remaining solution was diluted with water, filtered, and the residue washed with dilute nitric acid and hot water. This insoluble residue was now treated with dilute *aqua regia* as described under the first method for the separation of the insoluble iridium.

	20 per Cent. Iridium Alloy.	25 per Cent. Iridium Alloy.
Experiment 1 ...	0·1030 grm. = 20·60% iridium	0·1266 grm. = 25·32% iridium
„ 2 ...	0·1020 „ = 20·40% „	0·1264 „ = 25·28% „

The solution from the above separation contains the platinum and a little lead. It was evaporated down to expel excess of nitric and hydrochloric acids, so that on cooling the platinic chloride just solidified. This was dissolved in water, and a few drops of sulphuric acid added to precipitate the lead, then diluted, and lead sulphate filtered off. The platinum was then precipitated with zinc, and treated as described under the first method.

The following are the results obtained:

	20 per Cent. Iridium Alloy.	25 per Cent. Iridium Alloy.
Experiment 1 ...	0·3965 grm. platinum = 79·30%	0·3726 grm. platinum = 74·52%
„ 2 ...	0·3980 „ = 79·60%	0·3730 „ = 74·60%

Summary of results:

Approximate Composition.			By Silver Method.	By Lead Method.
		Per Cent.	Per Cent.	Per Cent.
Alloy 4.	Iridium	20	20·20 20·40	20·60 20·40
„	Platinum	80	79·76 79·52	79·30 79·60
Alloy 5.	Iridium	25	25·20 25·24	25·32 25·28
„	Platinum	75	74·68 74·56	74·52 74·60

In general practice with these alloys it is usual to determine the iridium only and to take the platinum by difference.

* * * * *

THE CHANGES IN THE CHARACTER OF FATS DURING THE PROCESS OF COOKING.

By HELEN MASTERS and HENRY LLEWELLYN SMITH.

(*Read at the Meeting, June* 3, 1914.)

But little work appears to have been done on the subject of changes in fats during the process of cooking. Various books on dietetics state that fats are somewhat hydrolysed on cooking, but no other changes appear to be mentioned. Fulmer and Manchester (*J. Amer. Chem. Soc.*, 1908, **30**, 1477) show that on heating cotton-seed oil between the temperatures of 180° and 270° C. there is a gradual and slight increase in the refractive index, a small decrease in the iodine value, and, above 220° C., a decided increase in acidity; the saponification value appears to change but little.

In the analysis of foods, it appears of interest to know to what extent the analytical constants of fats may be changed during the process of cooking, and the knowledge may also be of some dietetic interest.

In the present paper the authors have confined the experiments to the changes which occur when fats are cooked with flour.

Preliminary experiments with butter-fat showed but little change as measured by the refractive index and the iodine value, though it must be pointed out that the natural fat of the flour would raise the iodine value about a unit in the proportion used:

	Iodine Value.	$[N]_D$ 40° C.
Original butter-fat	38·3	1·4542
Extracted from pastry A ...	38·4	1·4543
" " B ...	38·0	1·4543

Similar experiments with cotton-seed oil showed greater changes:

	Iodine Value.	$[N]_D$ 40° C.	Acidity: Mgrms. KOH per Grm.
Original oil	104·4	1·4642	1·4
Extracted from pastry A	104·8	1·4643	—
" " B	103·9	1·4643	1·4
" " C	96·3	1·4652	2·1
" " D	98·5	1·4641	1·6

Refined cotton-seed oil was used in a number of experiments, as being more unsaturated, greater changes were to be expected, and it was hoped by a study of these to show the general type of changes to be expected when fats are cooked with flour; moreover, as some fats are now prepared from this oil for culinary purposes, the figures may be of interest.

In the following experiments, flour 200, cotton-seed oil 60, and water 50, or flour 200, butter-fat 100, and water 80, were mixed as pastry, without the addition of baking-powder; the mixing was always done in the same way, so as to make the experiments comparable. The flour used was a "pastry whites," and appeared to be a normal unbleached flour, containing no nitrite or persulphate. It contained 1·2 per cent. of fat which had an iodine value 110, $[N]_D$ 40° C. 1·4781, and an acid value 2·6 mgrms. KOH per grm.

The temperature of the oven was about 200° C., but the interior of the thicker pastries did not reach a higher temperature than 100° C. Controls of fat heated in the oven at the same time naturally reached the temperature of the oven. The time of cooking was varied, in order to obtain progressive changes.

After baking, the pastry was broken up and the fat extracted with ether; the ethereal extract was then filtered, and the ether distilled off.

A control experiment was carried out by mixing flour and oil in the same proportion as above, but without water, and extracting after about an hour. The oil showed no change in the iodine value nor in the refractive index; neither could oxidised acids be found, whilst they were always present in the fats extracted from the pastries. The acid value, however, had increased from 1·0 to 1·5.

The fats were also heated alone at 200° C. for an hour while oxygen was passed through; the analytical figures for these are compared with the extracted fats below.

ANALYSES OF COTTON-SEED OIL.

	Iodine Value.	$[N]_D$ 40° C.	Acidity Mgrms. KOH per Grm.	Saponification Value.	Oxidised Acids.	Apparent Acetyl Value.	Volatile Acids: Mgrms. KOH per Grm.	True Acetyl Value.
					Per Cent.			
Original oil	106·5	1·4649	1·0	194·0	Nil	11·0	2·4	8·6
Pastry 1	104·5	1·4651	2·0	196·5	0·45	16·4	3·9	12·5
" 2	103·1	1·4657	2·3	196·0	0·82	28·0	8·7	19·3
" 3	106·3	1·4654	1·0	197·6	0·15	16·2	4·9	11·3
" 4	103·8	1·4655	1·4	198·5	0·21	20·8	5·0	15·8
" 5	100·0	1·4667	1·5	202·0	2·30	24·0	4·2	19·8
" 6	100·0	1·4667	1·3	198·6	0·89	21·5	5·2	16·3
" 7	89·3	1·4674	2·0	205·4	4·15	32·3	9·1	23·2
Oil heated in oven, 30 minutes... ...	97·5	1·4652	0·8	197·3	0·12	11·7	3·4	8·3
Oil heated in flask, one hour, 200° C., oxygen passed through ...	95·9	1·4673	2·0	198·6	3·12	30·0	5·4	24·6

Pastries 3, 4, and 5 were the same, but cooked for different times, 3 being lightly cooked and 5 considerably overcooked.

Pastries 6 and 7 were the same batch, 7 being overcooked. These two were much thinner than the others.

ANALYSES OF BUTTER-FAT.

	Iodine Value.	$[N]_D$ 40° C.	Acidity Mgrms. KOH per Grm.	Saponification Value.	Oxidised Acids.	Reichert-Meissl No.	Apparent Acetyl Value.	Volatile Acids: Mgrms. KOH per Grm.	True Acetyl Value.
					Per Cent.				
Original butter-fat	31·8	1·4536	0·8	236·5	Nil	30·4	50·9	46·1	4·8
Pastry 1 ...	31·5	1·4540	0·9	236·4	0·12	29·8	52·4	46·8	5·6
„ 2 ...	31·8	1·4540	1·0	234·2	0·08	29·9	53·3	47·3	6·0
„ 3 ...	30·9	1·4544	1·5	238·3	0·24	30·3	54·1	46·5	10·6
Fat heated in oven, 30 minutes ...	30·1	1·4543	0·8	234·3	0·06	30·9	51·9	47·4	4·5
Fat heated in flask, one hour, 200° C., and oxygen passed through ...	26·8	1·4550	1·9	237·1	0·75	31·4	57·2	47·3	9·9

The three pastries were made at the same time. No. 1 was about three times as thick as the other two. Nos. 1 and 2 were cooked for the same time. No. 3 for twice as long.

The results show as the more obvious changes a decrease in the iodine value, an increase in the acetyl value and in the refractive index, and a slight increase in the acidity. From these it seems that the first change may be the hydroxylation of the unsaturated molecule. No definite ratio between the decrease in the iodine value and the increase in the acetyl value can, however, be traced; some oxidation of the fat also takes place, as shown by the increase of viscosity and darkening in those cooked for longer times, especially when the pastries were thin. "Oxidised" acids are formed, and in some cases these were estimated. These "oxidised" acids, described by Lewkowitsch ("Chemical Technology of Oils and Fats," 1 and 65, ANALYST, 1899, **24**, 325) are characterised by being insoluble in petroleum ether, but soluble in ethyl ether. They were not present in the original fats, but were always present in the cooked fats. The iodine value of the "oxidised" acids from one pastry made with cotton-seed oil was 64·7, whilst from the blown oil these acids had an iodine value of 59·2. The increase in the volatile acids may be partially due to the presence of these "oxidised" acids, as Lewkowitsch states that they may break up and yield volatile acids when heated in a current of steam. One sample obtained from pastries was distilled in a current of steam under the same conditions as were the volatile acids; volatile soluble acids were obtained equivalent to 3·5 mgrms. KOH per grm. of acid; this is insufficient to account for the increase in the volatile acids.

The changes generally are of the character which occur in the blowing of oils, and the same irregularities may be observed, the various constants not changing together.

The changes appear, however, to take place more readily in pastry, probably owing to surface action.

In all cases the cotton-seed oil extracted from pastry gave a positive reaction with Halphen's reagent; the reaction was also given by the oil which had been heated alone, but the blown oil gave only a slight reaction, and this only after some time.

The changes in the butter-fat are of a similar character, but somewhat less in degree.

It is not possible to make any definite statement as to the mechanism of the changes. Hydrolysis certainly does not take place to any very great extent. The increase in the acetyl value may be due to the formation of mono- or di-glycerides, but the small increase in acidity does not support this view.

The changes in a fat cooked normally are evidently but slight, unless the pastry is very thin; it is only in such thin pastries, or when they are considerably overcooked, that any appreciable change takes place in the ordinary analytical constants. There would be no great difficulty in detecting adulteration or substitution in most cases.

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ON FRESENIUS'S METHOD FOR DETERMINING SMALL QUANTITIES OF NITRITES, AND ITS SENSITIVENESS COMPARED WITH THE *m*-PHENYLENE-DIAMINE REACTION.

By E. A. LETTS and FLORENCE W. REA.

In the seventh (English) edition of Fresenius's "Quantitative Analysis," vol. ii., under Analysis of Potable Waters, a method is described in which nitrites are determined by a colorimetric reaction carried out in glass cylinders, depending upon the depth of the blue tint and the time which elapses before the production of the latter, when a potable water containing a nitrite is mixed with a solution of zinc iodide and starch, being afterwards acidulated with dilute sulphuric acid.

To prepare the zinc-iodide-starch solution, the directions given are—to boil 5 grms. starch and 20 grms. zinc chloride with 100 c.c. distilled water for several hours, the water being replaced as it evaporates, until the starch granules are nearly all dissolved; 2 grms. zinc iodide are then added, the solution diluted to 1 litre and filtered.

It is stated that the filtration is slow, but that a clear liquid is obtained, which, after some months, deposits a few flocks, but remains colourless if kept in a well-closed bottle in the dark (Richter, *Zeitschr. f. anal. Chem.*, 1870, 8, 358).

We have twice prepared this solution, but have found the filtration to be very slow, and it is better in our opinion to allow the solution to settle for some weeks, and to decant quantities of the clear liquid as required.

Fresenius recommends the employment of a solution of potassium nitrite, 1 c.c. of which contains 0·01 mgrm. NO_3 (old notation), and describes the preparation of such a solution from the commercial nitrite by titration of its solution with permanganate and dilution to the proper volume.

0·01 mgrm. N_2O_3 (new notation) is equivalent to almost 0·0037 mgrm. N_2.

The further directions given by Fresenius when employing the test are to add to 50 c.c. of the water to be examined 1 c.c. of the starch mixture and 1 c.c. of dilute sulphuric acid, and to compare, first, the time required before a blue tint begins to appear, and, secondly, the depth of this blue tint with those obtained with known volumes of the standard nitrite solution, added to 50 c.c. of distilled water and the same volumes of starch solution and sulphuric acid after given intervals of time.

Instead of employing only 1 c.c. of the starch solution, we have found that 4 c.c. gives better results. Also Fresenius does not state the strength of the dilute sulphuric acid which he employed. The strength which we have used is that of 1 vol. of the pure acid to 5 vols. of water, and of this mixture we have employed in each determination 2 c.c. instead of the 1 c.c. recommended by Fresenius.

Two solutions of the starch mixture were made by us, one about a year ago, and the other only a few weeks ago. For some reason which we have not as yet ascertained, the first of these solutions has been found to be much more sensitive than the second.

The standard solution of nitrite which we have employed was one of the pure sodium salt, and was so made as to contain 0·005 mgrm. nitrous nitrogen per c.c.

The following are some of the results obtained:

Volume of Nitrite Solution taken.	= Mgrm. Nitrogen.	Time when Blue Colour first appeared.		
C.C.		(1)	(2)	(3)
0·05	0·00025	7 minutes.	8 minutes.	8 minutes.
0·10	0·0005	3 ,,	3½ ,,	4 ,,
0·20	0·0010	1 minute.	1½ ,,	—
0·30	0·0015	Almost immediately.	Almost immediately.	Almost immediately.

Bright light has an undoubted effect on the rapidity with which the blue colour develops. Thus when the above results were obtained, and when the older starch solution was used, the day was very bright and sunny, and the contents of a Nessler cylinder containing 50 c.c. distilled water plus 4 c.c. starch solution and 2 c.c. dilute sulphuric acid began to acquire a slight blue colour in fifteen minutes; whereas in the following results, in which the newer starch was used, and when the day was dull and gloomy, a similar blank experiment remained without any colour appearing for upwards of an hour.

In order to compare the following results with those obtained by the *m*-phenylenediamine reaction, which is usually employed for determining small amounts of nitrites, some of the reagent was prepared (0·5 per cent. solution acidulated with dilute sulphuric acid).

Volume of Nitrite Solution taken.	= Mgrm. Nitrogen.	Time when Blue Colour first appeared.
C.C.		
0·05	0·00025	28 minutes.
0·10	0·0005	18 ,,
0·20	0·001	7 ,,
0·30	0·0015	3 ,,

In the results which follow, 1 c.c. of this reagent and 2 c.c. dilute sulphuric acid (1 : 5) were added to the volume of the nitrite in question contained in a Nessler cylinder, and the mixture made up to 50 c.c. with distilled water and well agitated.

Volume of Nitrite Solution taken.	= Mgrm. Nitrogen.	Result.
C.C.		
0·1	0·0005	Nothing after 30 minutes.
0·2	0·001	Possible slight darkening after 30 minutes, but not decided.
0·3	0·0015	Ditto.
0·5	0·0025	Slight colour in 3 minutes.
1·0	0·005	Well-marked colour in 6 minutes.

From the above results it would seem that 0·05 c.c. is the limit of sensitiveness of the zinc-iodide-starch method, and possibly 1 c.c. that of the *m*-phenylene-diamine reaction, and if that be the case, the former is twenty times as sensitive as the latter.

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Electrical Conductivity of Beers. H. Lüers. (*Zeitsch. ges. Brauwesen*, 1914, **37**, 210-213, 227-230; through *Chem. Zentralbl.*, 1914, I., 2069-2070.)—In the electro-chemical titration of beers with barium hydroxide by the method of Emslander, the curve rises steadily until the point of approximate neutrality to litmus is reached, and then begins to waver and to fall owing to the separation of phosphates. After all the phosphate has separated, the curve forms an arch, and finally rises in consequence of the appearance of free OH and Ba ions. Each beer has a characteristic form of curve depending upon the nature of the raw materials. Thus Pilsener beer shows a long, drawn-out curve; Munich beer a compressed form; and Kulmbach beer a broad curve, owing to its high proportion of constituents from high-coloured malts. In many of the North German beers the curve falls sharply at first, instead of rising, owing to the high proportion of sulphates. The lower turning-point of the curve (where all the phosphates have been converted into insoluble phosphates) may be ascertained exactly by treating the beer beforehand with pepsin, or prolonging both lines to the point of intersection. Here pH = 8·8 to 9·2. A sour beer that has been restored to its normal degree of acidity gives a different curve from that of the original beer, so that in this way it may be possible to detect the presence of neutralisation agents. C. A. M.

Estimation of Camphor in Tablets and Pills. E. Dowzard. (*J. Ind. and Eng. Chem.*, 1914, **6**, 489-490.)—The camphor is expelled from the tablets, etc., by means of a current of steam, and the distillate, containing the dissolved and undissolved camphor, is extracted with benzene. A specially wide form of condenser is necessary to prevent the camphor blocking the tube. From the optical rotation of the benzene solution the amount of camphor may be calculated. Average estimations with several samples of camphor showed that each 0° 1′ of rotation corresponded to 0·01961 grm. in 50 c.c., or 0·0098 grm. in 25 c.c., of the benzene solution. The factor for each apparatus and mode of working should be ascertained by estimations with known weights of camphor. C. A. M.

Some Reactions of Chrysophanic Acid, with Reference to its Detection in Complex Medicinal Preparations. E. M. Bailey. (*J. Ind. and Eng. Chem.*, 1914, **6**, 320-321.)—Chrysophanic acid, dioxymethylanthraquinone ($C_{15}H_{10}O_4$), occurs notably in senna and rhubarb, and may also be obtained from anthranol chrysarobin ($C_{15}H_{12}O_3$) present in Goa powder. It is soluble in alkalis, forming a deep cherry-red colour, and is yellow in acid solution, but has not been investigated as an indicator. Towards reactions involving oxidation it appears to be stable, and when taken into the body is apparently eliminated unchanged in the urine. In the investigation of patented and other medicinal preparations, the detection of chrysophanic acid serves to indicate the presence of a restricted group of vegetable constituents, and the similarity of many of its reactions to phenolphthaleïn makes its detection in presence of that substance desirable. When an alcoholic extract containing chrysophanic acid is dealcoholised or sufficiently diluted with water, acidified with a few drops of strong hydrochloric acid, and extracted with ether (acetone being used to destroy any emulsion formed) the colouring matter is taken up by the solvent, and the ethereal layer is coloured yellow. On washing the solvent with dilute alkali, such as 2·5 per cent. ammonia, the colour is transferred to the aqueous solution, which is coloured red. Picric acid and the colouring matter of hydrastis do not behave thus when treated with alkali, nor does their presence interfere with subsequent tests, but curcumin, hæmatoxylin, and phenolphthaleïn yield colours that may be indistinguishable from those given by chrysophanic acid. In the cases of curcumin, and hæmotoxylin, the colour of their alkaline solutions slowly fades (the red colour disappearing at 40°-50° C. in several hours), but that of chrysophanic acid and phenolphthaleïn is persistent. The interfering colour principles of curcumin and hæmatoxylin may be eliminated by acidifying the solution and shaking out with ether, when the ether extract may then be shown to be free from them. Phenolphthaleïn may be eliminated by transferring a portion of the ether extract just mentioned to a test-tube, driving off the ether, and adding a little zinc dust, together with 4 or 5 c.c. of 25 per cent. sodium hydroxide. On boiling, the red colour is discharged, the chrysophanic acid being reduced, leaving a yellowish colour, and the phenolphthaleïn is reduced to phthalin, which is colourless. If the alkaline solution is now diluted or a little hydrogen peroxide is added the characteristic cherry-red colour of chrysophanic acid is again produced, but phthalin remains unoxidised.

Curcumin and hæmotoxylin are both reduced by zinc dust and caustic soda, and are not subsequently oxidised by hydrogen peroxide, but their yellow or brown solutions mask the colour of chrysophanic acid. It is therefore preferable to eliminate them by the method described above. H. F. E. H.

Study of Methods of Extraction by Means of Immiscible Solvents from Point of View of Distribution Coefficients. I. (Analysis of Vanilla Extracts and Extraction of Acetanilide, Saccharin, and Caffeine.) J. W. Marden.

(*J. Ind. and Eng. Chem.*, 1914, **6**, 315-320.)—Several well-established "shaking-out" methods have been examined, with the result that some have been found to be less exact than was supposed, but to be readily capable of improvement, whilst others were found to be unnecessarily wasteful of solvents. For the estimation of acetanilide in hydrogen peroxide solutions (*Bulletin* 150 of the U.S. Bureau of Chemistry) directions are given to shake the solution twice with ether and once with chloroform and to evaporate the combined extracts, but no reason is given for the use of two solvents nor any suggestion made as to quantities. The authors of the bulletin state that the method extracts 95 per cent. of the acetanilide. This is the case if 20 c.c. portions of solvent are used to extract 50 c.c. of the solution, but if chloroform be used for each extraction 98·5 per cent. may be extracted. By using smaller portions of solvent at a time an equal volume may be made to give an even better result, or an equally good result may be obtained with economy of solvent.

For the extraction of saccharin from aqueous solutions (*Bulletin* 107 of the U.S. Bureau of Chemistry) directions are given to render the solution slightly acid with hydrochloric acid, and to extract with ether "as in the case of salicylic acid." The cases could hardly be less alike, viewed from the point of the respective distribution ratios. Salicylic acid may be almost quantitatively extracted with ether by a few washings, whereas, from very faintly acid solutions, four washings with 20 c.c. portions of ether will not extract as much as nine-tenths of the saccharin from 100 c.c. A better solvent was found in amyl alcohol, of which four 10 c.c. portions will extract 99·9 per cent. of saccharin from 50 c.c. of an acid aqueous solution. Amyl alcohol, however, being somewhat unpleasant to work with, other methods were tried, and it was found that the ether method was greatly improved by rendering the aqueous solution strongly acid (5 c.c. concentrated hydrochloric acid per 100 c.c.). Under these conditions, four 20 c.c. portions of ether were found to extract 99·5 per cent. of the saccharin from 100 c.c. of aqueous solution.

In his method for the estimation of tannin in tea, Smith (ANALYST, 1913, **38**, 312) removes caffeine from 50 c.c. of aqueous extract by treatment with four successive portions of 30 c.c. of chloroform. It is shown that four portions of 10 c.c. would remove 99·7 per cent. of the caffeine.

Experiments are described showing the distribution of vanillin, coumarin, and acetanilide between water and ether on the one hand, and between dilute ammonia and water on the other, and Hess and Prescott's method for the analysis of vanilla extracts and Winton and Bailey's modification of it are discussed in the light of the results so obtained. The question arises whether Winton and Bailey's modification is an improvement, but the adverse evidence is not pressed in view of the complexity of vanilla extracts and the general approval given to Winton and Bailey's method by competent critics. (For references to these methods, *cf.* ANALYST, 1913, **38**, 206.)

G. C. J.

Aroma of Hops. Study of the Volatile Oil with Relation to the Geographical Sources of the Hops. F. Rabak. (*J. Agric. Research*, 1914, **2**, 115-159.)—The samples, which were taken over a period of years and include both fresh and cold stored hops, were all of American origin with the exception of some imported Saaz. Amounts varying from 50 to 200 pounds were subjected to steam distillation, the yield of volatile oil varying from 0·192 per cent. (New York) to 0·370 per cent. (Washington), the average value of all the samples examined from 1906 to 1909 being 0·311 per cent. When distilled for a period of more than two seasons, all the hops showed considerable fluctuation in the yield of oil, and the order of their average yield is as follows: Washington, Californian, Saaz, Oregon, and New York. Determinations were made of the acid, ester, and saponification values of the oil from different samples of hops. The esters are considered to be the odour bearers, and considerable weight is attached to their estimation, which is made by saponifying the oil with alcoholic alkali and calculating the number of mgrms. of potassium hydroxide consumed in the reaction by 1 grm. of the oil. The acid number is determined by direct titration, and is expressed by the number of mgrms. of potassium hydroxide required to neutralise the free acidity of 1 grm. of the oil. The saponification number is the approximate sum of the acid and ester numbers. The following are average values for the years 1906 to 1910:

HOP OIL CONSTANTS.

Source of Hops.	Acid Number.	Ester Number.	Saponification Number.
Californian (average of five samples)	1·46	45·6	47·0
Oregon	2·70	56·0	61·5
New York	3·75	50·9	54·2
Washington	1·75	52·8	54·0
Saaz	2·02	23·5	21·0

Nine other samples of foreign (*i.e.*, not American) hops showed that the ester number of their hop oil was uniformly lower than the native material, values such as 15·7, 21·3, and 18 being found. That the unusually low ester content of the European hops is responsible for their generally acknowledged superior aroma cannot be positively stated, although it is highly probable, since it is known that other oils with a low ester content—notably lavender and peppermint oils—are more agreeable and fragrant than oils with a high ester number. A large part of the author's work is concerned with an account of the detailed fractional distillations of hop oils from different sources, for which the original paper must be consulted, the main conclusions being as follows:

The volatile oil of hops is shown to consist chiefly of the hydrocarbon myrcene, the heptoic, octoic, and nonoic esters of the alcohol myrcenol, and the sesquiterpene humulene with traces of free acids, formaldehyde, and probably some free alcohols. Of these the esters, which constitute a large proportion of the oil, are the most important. The curves of fractional distillation, which may be said to represent a partial quantitative separation, bring out strongly the relationship which exists between the hops from any source during one or several seasons. This relationship is also seen in the optical rotation curves of the oils. In general the physical properties of the oils show the strong similarities which may exist among related oils, and *vice versa*. The low ester value of the imported hops is a constant feature from year to year. It is clear that the geographical source of hops may be indicated by the ester number of the distilled oil, since experiments show that such numbers from hops of any particular source or season are very similar. H. F. E. H.

Malt Extract. H. T. Graber. (*J. Ind. and Eng. Chem.*, 1914, **6**, 403-404.)—Concentrated glycerol extracts of malted barley, which, owing to the lapse of time, had acquired an almost black colour and a disagreeable taste, were found still to have their original starch converting power (diastatic activity) when not more than a year old, after which time their enzymic activity greatly diminished, owing probably to the increase in acidity, which may reach a figure as high as 4·5 per cent., calculated as lactic acid. This developed acidity is also responsible for the marked blackening of the older extracts. Figures are given showing the composition of different extracts whose ages vary from one to thirty-two months, it being assumed that the whole of the maltose and dextrose is extracted in a Soxhlet apparatus by means of absolute alcohol, together with some colouring matters and glycerol. Dextrin and proteins are estimated in the aqueous extract of the residue. H. F. E. H.

Nephelometric Method for the Determination of Fat in Milk. W. R. Bloor. (*J. Amer. Chem. Soc.*, 1914, **36**, 1300-1304.)—The principle involved is the comparison of the light-reflecting power of an aqueous suspension of the milk fat with that of a similar suspension of a standard fat (triolein).* The method has the advantage of being applicable to a very small amount of milk—1 c.c. as a rule—and the time required is no more than that taken by the Gerber method. One c.c. of the milk is run slowly, with stirring, from an accurate pipette into about 80 c.c. of an alcohol-ether mixture (3 parts redistilled alcohol and 1 part of redistilled ether) in a 100 c.c. graduated flask. The measurement may be checked by weight. The mixture is then raised to boiling by immersion in a boiling-water bath, then cooled, made up to 100 c.c. with the alcohol-ether mixture, shaken, and filtered. The filtrate should be clear and almost colourless. Five c.c. of this solution are then run slowly and with stirring from a pipette with its point submerged into 100 c.c. of distilled water in a beaker. A similar solution is prepared with 5 c.c. of the standard fat solution, which contains about 2 mgrms. of triolein in every 5 c.c. of distilled absolute alcohol and pure dry ether. To the standard and the test solutions are simultaneously added 10 c.c. portions of 10 per cent. hydrochloric acid, and, after stirring, the solutions are allowed to stand five minutes, after which they are transferred to the comparison tubes of the nephelometer.

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* The aqueous suspension is obtained by flocking a colloidal solution of the fat with an electrolyte. Hydrochloric acid is the most suitable. The colloidal solution is produced by mixing an alcoholic solution of the fat with distilled water. Fat in blood has been determined by the author in a similar manner (Bloor, *J. Biol. Chem.*, 1914, **17**, 377).

At least five observations should be taken alternately from above and below, and the average taken. This reading is corrected from the calibration curve of the instrument, and the fat value of the milk calculated from this. The Richards nephelometer, made by the International Instrument Company, Cambridge, Mass., U.S.A. (Bloor, *J. Biol. Chem.*, 1914, **17**, 377), may be used, or a colorimeter such as the Duboscq may be adopted (ANALYST, 1913, **38**, 571). Since the amount of light reflected is not exactly proportional to the depth and fat value of the solutions, it is necessary to calibrate the nephelometer for different strengths of test solutions and for different standards if more than one are used. The values so obtained are plotted on a curve and corrections in the readings made from it. It was found that duplicate determinations made on the same milk agreed within the limits of error of the instrument (about 3 per cent.). H. F. E. H.

Fat Content of Milk and Rate of Coagulation with Rennet. A. Kreidl and E. Lenk. (*Biochem. Zeitsch.*, 1914, **63**, 151-156.)—The time required for the coagulation of a milk with rennet is not constant even under similar experimental conditions. The rate of coagulation varies with the fat content, and is smaller the richer the milk is in fat. E. W.

Identification of Traces of Strychnine by means of Manganese Carbonate. G. Guérin. (*J. Pharm. Chim.*, 1914, **9**, 595-597.)—The author states that the potassium dichromate reaction for strychnine does not give the blue coloration with less than 0·001 mgrm. of the alkaloid. Mandelin's reagent (0.5 grm. ammonium vanadate in 100 grms. of sulphuric acid) and Wenzell's reagent (0·5 grm. potassium permanganate in 100 grms. of sulphuric acid) give better results, and the colorations are less fugitive, whilst Sonnenschein's test (adding cerium oxide to a solution of the alkaloid in 2 or 3 drops of sulphuric acid) is still more sensitive. A reaction of great sensitiveness is produced by dissolving the alkaloid residue in 2 to 3 drops of sulphuric acid and adding 3 mgrms. of manganese carbonate. On stirring the liquid, a blue coloration, changing to violet and then to rose, is obtained in the presence of strychnine. C. A. M.

Separation and Estimation of Sugars in Gingerbread and Similar Products. J. König and W. Burberg. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1914, **27**, 761-774.)—A quantity of 6·25 grms. of the powdered sample is mixed in a 250 c.c. flask with 25 c.c. of water, and quantities of 50 c.c. of 96 per cent. alcohol are added from time to time over a period of two hours, until the flask is filled to the mark. The alcoholic solution is then filtered, evaporated to a small volume to remove the alcohol, and diluted with water to 250 c.c. Portions of this solution are used for the polarisation before and after inversion, the estimation of total sugars (residue on evaporation), dextrose and invert sugar (by Soxhlet's method), gravimetric estimation of the total invert sugar, and cane sugar. Starch and dextrins are estimated in the portion of the sample insoluble in alcohol. W. P. S.

Milling of Wheat and Testing of Flour. H. McCormack. (*J. Ind. and Eng. Chem.*, 1914, **6**, 423-428.)—The object of this paper is to urge the need for uniform methods to be employed in the laboratory for milling and testing operations, and for a uniform method of expressing the results obtained. Numerous analyses of wheats and flours are given, showing the variations in the results returned when the same sample was sent to different chemists, most of these differences were undoubtedly due to the varying methods employed, especially those for milling a sample of wheat. As it is impossible, on the laboratory scale, to reproduce factory conditions of grinding, bolting, etc., some method which is capable of being correlated with actual practice is essential, since the yield of patent flour from a sample of wheat will vary with different laboratory mills from 45 to 90 per cent. of the straight flour milled. The author describes in detail the methods adopted by him for laboratory milling, and also those for determination of ash, moisture, gliadin, baking test, absorption, etc. It is recommended that some form of mechanical kneading apparatus which can be kept at constant temperature should be used in all kneading operations, and that the colour be determined on the samples of flour, and not on the bread, by some such method as the Lovibond tintometer reading in standard units of colour, and not by a percentage system based on a standard flour. The paper contains a résumé of analytical work as applied to wheat and flour.

H. F. E. H.

Detection of Coal-Tar Dyes in Wines. F. Wohack. (*Zeitsch. landw. Vers.-Wesen Österr.*, 1913, **16**, 890-892; through *Chem. Zentralbl.*, 1914, **1**, 1976.)—The amyl alcohol extract from 20 c.c. of the wine is heated on a water-bath, and a wool-thread is immersed in the solution; should the wool be coloured pink, and the colour not be changed to green on treatment with ammonia, the presence of coal-tar colours is indicated. Cazeneuve's test may also be used; in this method the wine is treated with mercuric oxide to precipitate natural colouring matters, and the mixture is filtered; the filtrate is rendered slightly acid with hydrochloric acid, and, after any precipitated mercurous chloride has been separated by filtration, the liquid is heated with the addition of a few wool-threads until the dye has been fixed on the latter. W. P. S.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Assay of Digestive Ferments. H. T. Graber. (*J. Ind. and Eng. Chem.*, 1914, **6**, 402-3.)—In a previous paper (ANALYST, 1912, **37**, 57) the author pointed out that the use of potato starch caused the diastatic activity of a pancreatin preparation to show 30 per cent. higher than when maize starch was employed. He now emphasises the necessity of using strictly comparable samples of even the same starch if concordant results are to be obtained, the principal causes of variation being the acidity and the moisture content of the starch. Litmus as an indicator of neutrality is insufficiently sensitive, and the starch should be neutral, or at most but very faintly acid, to cochineal. The starch employed should be of an average moisture content, with a preference towards the use of anhydrous starch. A further precaution is to use a diluted solution of iodine at a constant temperature.

H. F. E. H.

Nephelometry in the Study of Nucleases. P. A. Kober and S. A. Graves. (*J. Amer. Chem. Soc.*, 1914, **36**, 1304-1310.)—The method described for estimating undigested nucleic acids consists of adding a precipitant to a dilute solution of the nucleic acids and estimating the resultant suspensoids nephelometrically. Yeast nucleic acid alone has been used in the present work. Proteins combine in slightly acid solution with nucleic acids to form artificial "nucleins." This reaction was found to be given by 1 part of yeast nucleic acid in 1,000,000 parts of water, and egg white was eventually found to be the most suitable protein for this purpose. The albumin is obtained from the white of eggs, which is considered to contain 10 per cent. of albumin. A 1 per cent. solution of egg albumin was made by roughly weighing the whites of from four to six fresh eggs, shaking well with $\frac{N}{10}$ acetic acid (0·5 c.c. per grm. of egg white), diluting with 10 volumes of water, allowance being made for the original acid, and filtering twice. Fifty c.c. of the clear 1 per cent. solution are placed in a flask, 5 c.c. acetic acid added, and made up to volume with distilled water. This 0·2 per cent. solution was used as the precipitant, and the nucleic acid solution is made by dissolving 0·1 grm. nucleic acid in 100 c.c. of distilled water. This solution is further diluted before using. One volume (10 c.c.) of nucleic acid (not stronger than 0·01 per cent.) is precipitated by running in from a pipette, with its point submerged, 2 volumes of the 0·2 per cent. albumin solution. After a gentle rotary shaking, the solution is ready for reading in the nephelometer. In most cases 0·007 per cent. nucleic acid was used as a standard. H. F. E. H.

Ash of Silicotic Lungs. J. McCrae. (*S. African Inst. for Medical Research*, 1913.)—The right lungs of six miners who had died from silicosis were examined, the figures given below being the average values found. For comparison, the figures for the right lung of a male, age thirty, who had never worked underground, are included:

	Normal Lung.	Average of Six Silicotic Lungs.
Wet weight	649·00 grms.	1556·0 grms.
Dry weight	76·00 „	237·0 „
Ash	3·77 „	15·4 „
Silica	0·55 „	6·1 „
Ash: Per cent. of dry matter	4·96	6·4
Silica: Per cent. of ash	14·7	39·6
Silica: Per cent. of dry matter	0·73	2·6

The high wet weight of the silicotic lung is not entirely due to the presence of a large amount of fibrous tissue, but also to its carrying an excess of serous fluid. The analysis of the ash is as follows:

	Normal Lung.	Average of Silicotic Lungs.
SiO_2 per cent.	14·71	41·4
Fe_2O_3 „	7·34	5·7
Al_2O_3 „	5·47	7·7
CaO „	10·98	3·6
MgO „	2·66	1·4
Na_2O „	18·31	10·0
K_2O „	10·04	5·6
SO_3 „	19·84 2·02	21·2 0·5

Since the lungs of infants contain no silica, it is probable that this substance is not normal to the healthy lung, and any found has gained access as dust during life. The author has therefore eliminated the silica found in both healthy and silicotic lungs, so as to be able to compare the other elements on a common basis. The figures as then calculated show that the alumina is higher in the silicotic lungs; an increase to be expected, since alumina accompanies silica in the dust inhaled. The proportion of lime is diminished in the diseased lungs, but the magnesia remains normal, as do the alkalis. The most striking feature is the marked increase in the proportion of phosphoric acid in the silicotic lungs, which is probably associated with the great production of fibrous tissue which occurs as silicosis proceeds.

In order to measure the size of the silica particles present in the diseased lungs, the organic tissues were destroyed by digestion with hydrochloric acid and potassium chlorate. Seventy per cent. of the residual particles were found to have a diameter less than 1 μ; the other 30 per cent. varied between 1 and 8·5 μ. H. F. E. H.

Detection of Guaiacol in Urine. O. Sammet. (*Zeitsch. Physiol. Chem.*, 1914, 91, 233-240.)—The phenolic bodies normally present in urine interfere with the reactions usually employed for the detection of guaiacol. Decomposition with hydriodic acid, followed by detection of the catechol or the methyl iodide formed was found to give the best results. The urine is made acid with 5 per cent. sulphuric acid, steam distilled, the distillate extracted with ether, and the ether extract dried over sodium sulphate and distilled. The residue is boiled for some time with concentrated hydriodic acid, using a reflux condenser, the mixture diluted with water, and the catechol formed extracted with ether. After evaporation of the ether the residue is tested for catechol by the usual methods. Even in the absence of catechol ferric chloride sometimes gives a faint greenish-brown colour, but this can never be mistaken for the intense emerald green colour produced when catechol is present: the change of colour to violet-red on addition of sodium carbonate does not occur in the absence of catechol. The reaction with ammoniacal silver nitrate solution is not so decisive, as some darkening is also produced in the absence of catechol. If the guaiacol is present in considerable quantity the catechol formed can easily be isolated in a pure form, and further identified by melting-point determination. For the detection of the methyl iodide formed on decomposing the guaiacol with hydriodic acid it is necessary to use petroleum ether or chloroform for the extraction of the steam distillate, since when ethyl ether is used a turbidity of the silver nitrate solution is produced in the absence of guaiacol. This is probably due to the retention of minute traces of ethyl ether, which reacts with the hydriodic acid, forming ethyl iodide. The distillation with hydriodic acid is carried out according to the usual Zeissel method. Attempts to estimate the guaiacol by weighing the silver iodide formed always gave low results (65-85 per cent.). This was found to be due to guaiacol being volatile to some extent in the vapours of petroleum ether and chloroform: this also accounts for the yields being smaller when larger quantities of solvent are used for extraction.

E. W.

Quantitative Estimation of the Oxyproteic Acid Fraction in Urine. R. Sassa. (*Biochem. Zeitsch.*, 1914, **64**, 195-222.)—The urine (200-1,000 c.c.) is evaporated to a syrup on the water-bath, with addition of a little acetic acid, and, after cooling, is made acid to Congo paper with sulphuric acid (20-25 c.c. 10 per cent. sulphuric acid per litre urine), and then treated with 3-4 volumes of 95 per cent. alcohol, filtered and washed with 75-80 per cent. alcohol. The filtrate is diluted with 2 volumes of water, an excess of finely powdered barium hydroxide added, and the excess of baryta removed by means of carbon dioxide. The mixture is concentrated to a small volume, filtered and washed with water free from carbon dioxide. The filtrate is evaporated to a thick syrup and ground with ignited kieselguhr to a moist powder, which is then mixed with 200 to 1,000 c.c. of a mixture of 1 part ether and 2 parts 95 per cent. alcohol and kept in a closed vessel for one day with frequent shaking. The mass is then filtered, washed with ether-alcohol, and ground to a fine powder, which is again extracted with absolute alcohol or a mixture of ether with absolute alcohol until the extracts are free from urea. The powder is then freed from the solvent, extracted with warm water, and the filtrate made up to a known volume. For the estimation of the proteic acid nitrogen, an aliquot portion of the filtrate is treated alternately with 20 per cent. mercuric acetate solution and

10 per cent. sodium carbonate solution until a permanent yellow or red precipitate begins to separate. This precipitate is collected and washed and the nitrogen estimated by the Kjeldahl method. E. W.

Gravimetric Estimation of Urea in Urine. R. Fosse. (*Comptes rend.*, 1914, **158**, 1588-1590.)—Ten c.c. of diluted urine (1 : 10) are treated with 35 c.c. of glacial acetic acid, and then with 5 c.c. of a 10 per cent. solution of xanthydrol in methyl alcohol, which is added in five separate portions at intervals of ten minutes. After one hour the precipitate is collected, washed with alcohol on a tared filter, and dried for a few minutes in the water-oven. The ureine thus precipitated contained 6·69 to 6·64 per cent. of nitrogen (theory 6·66 per cent.). No other nitrogenous substances in urine gave a precipitate with xanthydrol under these conditions.

C. A. M.

ORGANIC ANALYSIS.

Estimation of the Acetyl Value of Oils, Fats, etc. E. B. Holland. (*J. Ind. and Eng. Chem.*, 1914, **6**, 482-486.)—It is suggested that, in order to be readily comparable with other results in fat analysis, the acetyl value should indicate the number of mgrms. of potassium hydroxide required to saponify the acetyl taken up by 1 grm. of the fat on acetylation. It may be rapidly estimated by the following adaptation of several processes: Five grms. of the fat are heated with 10 c.c. of acetic anhydride in a boiling-water bath beneath a reflux condenser for one to one and a half hours, after which sufficient ceresin is added to give a solid disc when cold. Prior to cooling, 150 c.c. of boiling water are introduced, and the flask heated on the water-bath with occasional shaking to expel occluded acetic acid. The solid cake left on cooling is heated with a further 150 c.c. of boiling water, this process being repeated about six times until the filtrate is nearly neutral. The solid disc and particles on the filter are then boiled with 50 c.c. of standard alcoholic potassium hydroxide solution and 50 c.c. of alcohol beneath a reflux condenser (with glass beads to prevent bumping), and the excess of alkali titrated with $\frac{N}{2}$ hydrochloric acid, with alkali blue as indicator. The difference between the saponification value before and after acetylation is the acetyl value. C. A. M.

Differentiation of Natural and Oil Asphalts. E. C. Pailler. (*J. Ind. and Eng. Chem.*, 1914, **6**, 286-289.)—The author shows how difficult it is to distinguish between genuine Trinidad or Bermudez asphalts and mixtures of these with as much as 50 per cent. of petroleum pitch, when the examination is confined to the estimation of fixed carbon and mineral matter and the determination of the usual physical constants. The value of a sulphur determination, never great, has been much diminished since it has become customary to "sulphonate" petroleum products. The other tests, though insufficient alone, are useful, especially that for fixed carbon, by which the author does not mean the organic matter undissolved by carbon bisulphide, but the organic residue remaining after heating the asphalt in a covered crucible, as in the proximate analysis of coal.

The following tests, based on an observation of Marcusson (*Chem. Zeit.*, 1912,

36, 801) that, when natural asphalts are distilled, the first fractions contain notable amounts of organic acids and esters, have been found more useful than any other single tests. Such an amount of the material as contains 30 grms. bitumen is distilled from a 150 c.c. retort at the rate of 30 drops a minute, and the first and second 5 c.c. of distillate are each collected in a tared weighing-bottle. After weighing, the contents of each bottle are dissolved in 10 c.c. ether, any mineral acid which may be present is removed by shaking the ethereal solution with water in a separator, and the ethereal solution is then diluted with 75 c.c. neutral 95 per cent. alcohol and titrated with $\frac{N}{10}$ sodium hydroxide, using alkali blue as indicator.

The other test is a saponification test on the original asphalt, of which 5 grms. (ash-free) are dissolved in 30 c.c. benzene, and saponified by boiling with 50 c.c. $\frac{N}{1}$ alcoholic potash under a reflux condenser. After cooling, 250 c.c. of 95 per cent. alcohol are added, and the excess of alkali titrated with $\frac{N}{1}$ sulphuric acid, using alkali blue as indicator. The flask is heated again on the water-bath, and more acid added if the red colour returns. The end-point is distinct, in spite of the dark colour of the solution. Some results of these tests are appended. The saponification value is expressed in the ordinary way on the original (ash-free) substance, but the acid values represent mgrms. of KOH required to neutralise 1 grm. of the first and second fractions respectively of the distillate.

	Saponification Value.	Acid Values.	
		First Fraction.	Second Fraction.
Trinidad asphalt	40·0	16·00	8·20
Bermudez asphalt	28·0	9·70	3·40
Petroleum pitch: Californian ...	12·0	0·15	0·05
„ „ Mexican ...	10·5	0·23	0·07
Maltha flux	8·0	0·14	0·22

G. C. J.

Estimation of Cane Sugar in the Presence of Lactose. J. N. Rakshit.

(*J. Ind. and Eng. Chem.*, 1914, **6**, 307-308.)—The percentage of lactose in the solution is first determined by titration with Fehling's solution, and then a measured quantity of Fehling's solution is boiled with a calculated volume of the sugar solution, so as exactly to throw out all copper and decompose the whole of the lactose. In the filtrate the cane sugar is then determined in the ordinary way by acid inversion. Figures are given showing that, when working with known amounts of the pure sugars, the results only differ about 0·02 per cent. In milk preparations it is necessary first to precipitate the casein by the cautious addition of dilute citric acid, avoiding any excess, which renders the subsequent filtration difficult. Results of the same order of accuracy are then obtained as with the pure sugars.

H. F. E. H.

Estimation of so-called Free Carbon in Tar Products. J. M. Weiss. (*J. Ind. and Eng. Chem.*, 1914, **6**, 279-285.)—It is known that the so-called free carbon of pitch or tar is not elemental carbon, but a mixture of this substance with compounds of high carbon content, and the methods of estimation in use would only justify its definition as the material remaining undissolved when treated in a particular way with one or more solvents. Other things being equal, that solvent which leaves the smallest residue undissolved is to be preferred. Of the solvents for which such claims have been brought forward, however, only pyridine and aniline, among definite chemical substances, appear to give lower results than the benzene-toluene treatment described below, and the inconvenience attaching to the use of these solvents more than outweighs the very slightly improved results which they yield, pyridine being costly, difficult to purify, and objectionable to work with, and aniline solutions filtering with extreme slowness. Hooper's method (Warnes's "Coal-Tar Distillation," p. 146) is dismissed because it depends on the use of 90 per cent. benzol and cresylic acid, neither of which is of definite composition. The work of other investigators has pointed to the formation of an insoluble compound of some constituent of tar with carbon bisulphide when this is used as solvent, and the author provides quantitative evidence of this, as well as evidence that carbon tetrachloride, chloroform, and acetic acid are still more unsatisfactory solvents for a similar reason.

It is admitted that for asphalts in general the usual treatment with cold carbon disulphide gives results which cannot be improved on, and gives them more quickly than the method described below. That method, moreover, requires no more of the analyst's time, and gives results in close agreement with the carbon disulphide method when applied to asphalts. With tar products, on the other hand, the slow solution of the last portion of the bitumen makes it necessary to modify the carbon disulphide method at the sacrifice of its one merit—that of speed. Moreover, although some tar products yield almost identical results by either method, others—including those from vertical retorts—yield a much smaller residue when treated by the benzene-toluene method, which is therefore preferred.

Even toluene gives rise to the formation of notable amounts of insoluble matter if the solvent and tar product are left long in contact. The time given below for the preliminary extraction with toluene should therefore not be exceeded. From 5 to 10 grms. of the tar or pitch, according to its fixed carbon content, are digested for thirty minutes on the steam-bath with 50 c.c. of toluene, keeping the mixture well agitated. It is then decanted through a tared, dry thimble made of two thicknesses of Schleicher and Schüll's hardened paper (No. 575, 15 cm. diameter) supported in a carbon filter-tube, and the residue washed with hot toluene until the filtrate is colourless. The thimble is then transferred to a special extractor, figured in the text and obtainable from the A. H. Thomas Company, of Philadelphia, but substantially identical with that described by G. A. Stokes (ANALYST, 1914, 295). Here it is extracted with benzene until the solvent dropping from the thimble is colourless. The thimble is finally dried in the steam-oven, allowed to cool in a desiccator, and weighed in the weighing-bottle in which it was originally tared.

G. C. J.

Volumetric Estimation of Cyanamide. G. Grube and J. Krüger. (*Zeitsch. angew. Chem.*, 1914, **27**, 326-327.)—Kappen's method (*Landw. Versuchsstationen*, 1909, **70**, 445; see also ANALYST, 1911, **36**, 560), which depends on precipitation of cyanamide by an ammoniacal solution of silver nitrate and titration of the excess of silver by means of thiocyanate, has been criticised by Caro (ANALYST, 1911, **36**, 76) on the ground that the silver content of the precipitate varies with the experimental conditions, though the whole of the cyanamide is precipitated. In view of the latter fact, Caro proposed to estimate cyanamide by submitting the washed precipitate to Kjeldahl's process. Stutzer and Söll obtained identical results by both methods (*Zeitsch. angew. Chem.*, 1910, **23**, 1873) but Monnier alleged (ANALYST, 1911, **36**, 429) that both failed owing to the insufficient insolubility of the precipitate in ammonia. Of this, however, he adduced no evidence, and Stutzer showed (ANALYST, 1911, **36**, 429) that his results, all obtained by a modification of Kappen's method and none of them by Caro's method, could be equally well explained by the polymerisation of a portion of the cyanamide to dicyanodiamide under Monnier's experimental conditions.

The latter risk can be excluded in Kappen's method by adding the silver nitrate before the ammonia and in Caro's by rendering the solution faintly acid with acetic acid before adding the ammoniacal silver acetate. Under these conditions, the two methods yield identical and exact results, provided the concentration of ammonia and ammonium salts is small, but the precipitate is shown to be appreciably soluble in solutions of ammonia and ammonium nitrate. In applying Kappen's method, therefore, the solution is made acid with the least possible quantity of dilute nitric acid, diluted to 300 c.c., mixed with 50 c.c. $\frac{N}{10}$ silver nitrate, rendered just distinctly alkaline to litmus-paper by addition of $\frac{N}{1}$ ammonia, made up to 500 c.c. and filtered. The silver in 250 c.c. of the filtrate is then estimated by means of $\frac{N}{10}$ thiocyanate. The use of 20 c.c. of $\frac{N}{1}$ ammonia beyond what is necessary for neutralisation introduces an error of 2 per cent., which is also the error when an excess of 10 c.c. $\frac{N}{1}$ ammonia is added to a solution containing 20 c.c. of $\frac{N}{1}$ ammonium nitrate.

G. C. J.

Detection of Cyanides in Presence of Ferro and Ferricyanides and Thiocyanates. O. L. Burnebey. (*J. Amer. Chem. Soc.*, 1914, **36**, 1092-3.)—The reaction depends on the solubility of copper sulphide in solutions of alkali cyanides. A bubble or two of hydrogen sulphide is passed into a small quantity of copper sulphate solution ($\frac{N}{10}$ is a convenient strength). The suspension of copper sulphide is then added to the unknown ammoniacal solution (or *vice versa*) with constant shaking. If the colour disappears the presence of a cyanide is indicated. From the volume of copper solution used an approximately quantitative estimation can be made. In the presence of ferrocyanides the reaction is not quantitative, as the ferrocyanide is oxidised to ferricyanide. The colour caused by 0·1 c.c. of the copper solution in a volume of 5 c.c. is removed by 0·2 c.c. of $\frac{N}{100}$ solution of potassium cyanide, which means a delicacy of 1 part of HCN per 100,000. In the presence of 50 c.c. of a 4 per cent. solution of potassium thiocyanate, 0·5 mgrm. of HCN can be detected, 50 c.c. of a 4 per cent. potassium ferrocyanide allows of the detection of

1 mgrm. of HCN, while 0·5 mgrm. can be detected in 50 c.c. of a 4 per cent. solution of potassium ferricyanide. In the presence of small quantities of these compounds, the test is reliable to 0·1 mgrm. of HCN. H. F. E. H.

Estimation of Ferrocyanide by Titration with Alkali Bromate. L. L. de Koninck and N. Joassart. (*Bull. Soc. Chim. Belg.*, 1914, **28**, 144-148.)—Ferrocyanide can be accurately estimated by titration with $\frac{N}{10}$ alkali bromate, provided the acidity of the solution lies within certain somewhat narrow limits. The neutral solution of ferrocyanide is rendered strongly acid by addition of 20 c.c. of dilute hydrochloric acid (sp. gr. 1·095), and diluted to 100 c.c. with water before commencing the titration. The end-point is determined by means of a solution of iron alum, used as outside indicator. With the initial concentration of acid stated, any quantity of ferrocyanide up to that equivalent to 50 c.c. of $\frac{N}{10}$ solution may be estimated with an error not exceeding 0·1 c.c. of $\frac{N}{10}$ solution. If the final concentration of acid falls below 12 c.c. (sp. gr. 1·095) per 100 c.c., the results tend to be high, and are 30 per cent. too high when the concentration of acid falls to 10 c.c. per 100 c.c. High concentrations of acid also give rise to high results, the use of 30 c.c., instead of the 20 c.c. specified above, leading to an error of about 0·5 c.c of $\frac{N}{10}$ solution. With 25 c.c. of acid, the consumption of bromate solution is from 0·10 to 0·20 c.c. in excess of that required by theory. G. C. J.

Volumetric Estimation of Ferricyanide in Presence of Ferric Salts and Cyanides. E. Müller and F. Seidel. (*Zeitsch. anal. Chem.*, 1914, **53**, 416-420.)—Müller and Diefenthäler (ANALYST, 1910, **35**, 414) and also Mecklenburg (*ibid.*, 413) have shown that, though Mohr's original method for the iodimetric estimation of ferricyanides in alkali bicarbonate solution is faulty, by working in neutral solutions or solutions which are distinctly but not too strongly acid with a mineral acid, exact results may be obtained. The method breaks down, however, in presence of ferric salts, since these also liberate iodine from potassium iodide.

By taking advantage of the fact that ferric salts form, with alkali fluorides, insoluble compounds of the type K_3FeF_6 (*cf.* Greef, ANALYST, 1913, **38**, 521), the interference of ferric salts may be avoided. To the neutral or faintly acid solution of ferricyanide containing ferric salts, at least enough potassium fluoride is added to combine with all the ferric iron. Sufficient potassium iodide and 5 grms. crystallised zinc sulphate are next added, and after a few minutes the liberated iodine is titrated with thiosulphate, using starch as indicator. The results are exact.

In presence of hydrogen cyanide—always present in acid solutions of ferric salts and ferricyanide when these have stood some time—the above method breaks down, as does de Haen's method for the estimation of ferricyanide by reduction to ferrocyanide, and subsequent titration with permanganate. For, on attempting to reduce the ferricyanide with ferrous hydroxide in alkaline solution, as in de Haen's method, any hydrogen cyanide present is converted into ferrocyanide, and results above the truth are obtained. The unmodified iodimetric method above described, on the other hand, gives low results in presence of hydrogen cyanide, which reacts with iodine in accordance with the reversible equation, $HCN + I_2 = HI + ICN$, the extent to which the reaction proceeds from left to right depending on the hydrion concentration. Before ferricyanide can be estimated in solution containing cyanides, therefore, the solution must be rendered distinctly acid with a mineral acid, and hydrogen cyanide expelled by passing a current of carbon dioxide through the liquid, preferably heated to 50° C. If ferric salts are simultaneously present, alkali fluoride should be added before the expulsion of hydrogen cyanide, as, otherwise, Berlin green may be formed. G. C. J.

Method for the Estimation of Crude Fibre. H. Stiegler. (*J. Landw.*, 1913, **61**, 399-426; through *Chem. Zentralbl.*, 1914, I., 2016-2017.)—From 1·5 to 3 grms. of the finely powdered substance are mixed in a flask with 20 c.c. of water and 60 c.c. of hydrochloric acid (sp. gr. 1·19); after ten minutes 120 c.c. of boiling water are added, and the flask is immersed in a boiling-water bath for one hour, during which time a current of air is passed through the mixture. When the insoluble portion has settled the liquid is drawn off by means of a syphon, the shorter limb of which contains a small glass-wool filter plug; the latter is then blown into the flask, the acid residue is neutralised, 50 c.c. of a 5 per cent. potassium hydroxide solution are added, the mixture is diluted to 200 c.c. with boiling water, and heated in a water bath for thirty minutes while a current of air is blown through it. The crude fibre and glass-wool are then collected on an asbestos filter, washed with hot water, alcohol, and ether, dried at 100° C., and weighed. The filter and its contents are then ignited and again weighed, the difference between the two weighings giving the quantity of crude fibre. The results obtained agree with those found by Tollens' method. W. P. S.

Estimation of Halogens in Organic Compounds. M. Busch. (*Zeitsch. angew. Chem.*, 1914, **27**, 432.)—To the substance dissolved in alcohol are added a dilute solution of 0·2 grm. of sodium hydroxide, and 1 to 2 grms. of calcium carbonate coated with finely divided palladium (*Ber.*, 1913, **46**, 3069). The air is removed by means of a strong current of hydrogen, and the reaction completed in a shaking apparatus in an atmosphere of hydrogen under pressure. The halogen is determined by the usual methods in the filtrate from the catalyser, which may be used several times. O. E. M.

Estimation of Iodine in Organic Substances. R. Grützner. (*Chem. Zeit.*, 1914, **38**, 769-770.)—The essential features of the method described consist in igniting the substance in the presence of a peroxide, decomposing the excess of the latter with permanganate, and estimating the iodic acid volumetrically. The substance is mixed with sodium hydroxide, heated, and sodium peroxide or barium peroxide is added to the fused mass; a small quantity of charcoal may be added towards the end of the operation (which should not be too prolonged) to decompose the greater part of the excess of peroxide added. The cooled mass is then dissolved in water (sodium sulphate may be added when barium peroxide is used), the solution is filtered and boiled for a few minutes after the addition of potassium permanganate. The mixture is now acidified with sulphuric acid, again boiled, care being taken that a slight excess of permanganate is present, cooled, rendered alkaline, and boiled with the addition of a small quantity of alcohol. The hot solution is filtered, the filtrate heated to remove alcohol, and then rendered just acid to litmus by the addition of a mixture of phosphoric and sulphuric acids (3 volumes of phosphoric acid, 1 : 4, and 2 volumes of sulphuric acid, 1 : 4, treated with permanganate, and then decolorised with oxalic acid), solid ammonium carbonate is added, the mixture boiled for three minutes, cooled, rendered acid to Congo-red by the addition of sulphuric acid, treated with potassium iodide, and the liberated iodine titrated with thiosulphate solution. Six atoms of iodine are liberated for every atom of iodine present in the original substance. W. P. S.

Combustion Method for the Direct Estimation of Rubber. L. G. Wesson. (*J. Ind. and Eng. Chem.*, 1914, **6**, 459-462.)—From 0·25 to 0·5 grm. of the finely divided sample is extracted for three to four hours with boiling acetone, and the residue allowed to dissolve or swell up in 40 c.c. of warm chloroform, after which it is chilled and subjected to the action of nitrogen trioxide (prepared by dropping nitric acid of sp. gr. 1·3 upon arsenic trioxide contained in a flask warmed on the water-bath) until the chloroform retains a deep green tint for fifteen to twenty minutes. The next morning the rubber nitrosite is separated and washed with small quantities of chloroform (5 c.c.), and the excess of acid vapour and chloroform expelled by a current of air. It is then dissolved in acetone, the solution centrifuged to separate mineral matter, and an aliquot portion of the solution concentrated and transferred to a porcelain boat (14 cm. × 1·1 cm.) previously charged to about two-thirds of its capacity with alundum, while the flask is rinsed out with ethyl acetate. The boat is dried for two hours at about 85° C., and the nitrosite is then burned in a combustion tube in a current of oxygen, the products of decomposition being carried over a red-hot spiral of platinum wire to complete the oxidation. The oxides of nitrogen are absorbed by a saturated solution of potassium dichromate in strong sulphuric acid, while acid vapours and sulphur trioxide are retained by granulated zinc, and the carbon dioxide is received in a soda-lime absorption tube in the usual way. The completion of the combustion is indicated by finally passing the gas through a faint yellow solution of palladium chloride. The amount of carbon dioxide is calculated into $C_{10}H_{16}$. In the case of vulcanised rubber the sulphur has been found by Alexander (*Ber.*, 1907, **40**, 1077) to remain quantitatively in the nitrosite. If so it may be estimated in an aliquot portion of the acetone solution. C. A. M.

Estimation of Total Sulphur in India-Rubber. Utz. (*Gummi-Zeit.*, 1914, **28**, 631-632; through *Chem. Zentralbl.*, 1914, I., 2068-2069.)—The rapid method of Kaye and Sharp (*India-Rubber J.*, December 7, 1912) gives results sufficiently accurate for technical purposes, provided the oxidation be carried out at a low temperature, to prevent loss of sulphur. From 0·25 to 0·5 grm. of the finely divided sample of vulcanised rubber is mixed in a covered porcelain crucible with 8 parts by weight of zinc oxide and 4 parts of potassium nitrate, and heated first on an iron plate and then very gradually over a minute flame until the reaction is complete. The crucible is then heated for five minutes over a Bunsen flame, after which its contents are dissolved in hot dilute hydrochloric acid, and the sulphuric acid precipitated in the usual way. An estimation can be completed within two hours. C. A. M.

Relation between the Melting-Point and Viscosity of Refined Tars. P. P. Sharples. (*J. Ind. and Eng. Chem.*, 1914, **6**, 285-286.)—The rough dependence of the viscosity of refined tars on their melting-point has long been recognised. It is now shown that, for tars of the same fixed carbon content, the viscosity increases as the melting-point rises, but these two physical constants are not directly proportional. The viscosities of tars of the same melting-point, but of different carbon contents, increase with the carbon content. From a physical standpoint free carbon might be expected to impede the flow of the material, and its function in this direction, up to the point at which it tends to weaken the binding and lasting properties of the tar, affords a rational explanation of the preference shown by practical men for pitch containing neither more nor less than some assigned amount of fixed carbon, whether the pitch is to be used for briquetting or for road-making purposes. The inclusion of both the melting-point and viscosity in a physical examination of refined tar intended for use as a binder would seem to be warranted. G. C. J.

Composition of Water-Gas Tar. C. R. Downs and A. L. Dean. (*J. Ind. and Eng. Chem.*, 1914, **6**, 366-370.)—The gas-oil used in the Lowe plant, which produced the tar examined by the authors, was derived from Oklahoma petroleum. The tar appears to contain only traces of paraffins, as the fractions distilling below 250° C., with one exception, could be sulphonated up to 97 to 100 per cent. Moreover, the exceptional fraction (b.-pt. 180° to 205° C.) did not appear to consist of paraffins, since the unsulphonated matter was not a clear oil, but a putty-like substance with resinous odour, suggesting that it might be a polymerisation product of some unsaturated hydrocarbons.

Benzene (0·4 per cent.), toluene (1 per cent.), xylenes (1·2 per cent.), mostly with some zylene, mesitylene, naphthalene (8 per cent.), anthracene (0·4 per cent.), thiophene (0·01 per cent.) and higher homologues of thiophene, were isolated, and the opinion is expressed that water-gas tar could be worked up for the production of pure hydrocarbons or commercial products by methods similar to those employed with coal tar, and without encountering special difficulties. The original tar being substantially devoid of free carbon, the percentage of this in the pitch may readily be kept even lower than is usually specified in contracts for road-making or briquetting material. G. C. J.

INORGANIC ANALYSIS.

Estimation of Ammonia in Illuminating Gas. J. D. Edwards. (*J. Ind. and Eng. Chem.*, 1914, **6**, 468-469.)—The best indicator for this purpose is said to be sodium alizarinsulphonate, which is less sensitive to carbon dioxide than are cochineal or paranitrophenol, though both these indicators are fairly satisfactory. Methyl orange, though less sensitive to carbon dioxide than the indicators named, is said not to give sufficiently sharp colour changes with weak solutions. $\frac{N}{10}$ sulphuric acid is preferred to stronger acid as an absorbent, because of the small amount of ammonia to be absorbed and the consequent error introduced by a very small inaccuracy in titrating back a large excess of acid. A new form of absorption apparatus is figured, but it is agreed that the Referees' apparatus or many alternatives are equally satisfactory. With 5 grains per 100 cubic feet, an estimation within 10 per cent. is considered satisfactory; but with more ammonia present the error should be less, and with less than 1 grain per 100 cubic feet it may be greater. G. C. J.

Estimation of Arsenic in Hydrochloric and Sulphuric Acids. R. F. Farbell. (*J. Ind. and Eng. Chem.*, 1914, **6**, 400-401.)—The method described serves for the estimation of quantities of arsenic exceeding 1 mgrm. with an error in no case exceeding 5 per cent., whilst quantities down to 0·02 mgrm. may be estimated within 20 per cent. It depends on the evolution of the arsenic as arsine, the decomposition of the latter by means of a solution of iodine in petroleum ether with the production of arsenious iodide, which reacts with water to give arsenious oxide, this in turn being further oxidised by iodine to arsenic oxide. $As = 8I$. The apparatus employed consists of a 500 c.c. flask with two-hole stopper, one hole carrying a tap funnel, the other a Kjeldahl spray trap. The latter is connected to a Koeninck potash bulb, thence to a single bulb such as always precedes a Meyer's bulb tube, and finally to a Meyer's bulb tube. The latter is charged with 20 c.c. water, 50 c.c. gasoline purified as described below, and 50 c.c. of a solution made by dissolving 0·677 grm. of iodine in 1,000 c.c. of purified gasoline. The gasoline is purified by mixing with 5 to 10 per cent. of sulphuric acid, and agitating the mixture with air for 8 to 10 hours. The gasoline is then separated, allowed to stand over fresh acid for several days with occasional shaking, washed with dilute alkali, and finally with water. The potash bulb is charged with lead acetate solution to decompose any hydrogen sulphide, and about 35 grms. of zinc are placed in the flask. The acid to be tested, if concentrated, is broken down to half strength, and 50 c.c., or other convenient quantity, depending on its arsenic content, are run in through the tap funnel, and followed by 1 c.c. of 10 per cent. stannous chloride solution and a little water to wash the stem of the funnel. After two hours, about 20 c.c. of pure dilute (sp. gr., 1·4) sulphuric acid are run in, and the evolution of hydrogen allowed to proceed for another hour. The contents of the bulb tube are then transferred to a separator, the aqueous layer run off, and 50 c.c. of alkaline sodium arsenite solution, exactly equivalent to the iodine solution, are run into the separator and shaken. (The arsenite solution is made by dissolving 0·264 grm. arsenious oxide and 1 grm. sodium carbonate in hot water, cooling, adding 1 grm. sodium bicarbonate, and diluting to 1,000 c.c.) The aqueous layer is run off, mixed with starch solution, and titrated with a solution of iodine (0·677 grm. per litre) in potassium iodide. Each c.c. corresponds to 0·05 mgrm. arsenic. G. C. J.

Relation of Composition of Ash in Coal to its Fusing Temperature. O. W. Palmenberg. (*J. Ind. and Eng. Chem.*, 1914, **6**, 277-279.)—The author seeks to show that opinions as to the fusibility of coal ashes based on a consideration of the results of chemical analysis are worthless, and that a direct determination of the fusing-point is the only rational practice. Experimental results are given which exemplify the difficulty attending a prediction of the probable clinkering properties of a coal from an inspection of the results of the analysis of its ash. G. C. J.

Rapid Electrolytic Separation of Copper from Arsenic. A. Sieverts and W. Wippelmann. (*Zeitsch. anorg. Chem.*, 1914, **87**, 169-174.)—Ashbrook has stated (*J. Amer. Chem. Soc.*, 1904, **26**, 1285) that copper may be separated from an equal weight of arsenic either from ammoniacal or nitric acid solution, but does not state in what condition the arsenic was present in his experiments. Richardson (*Zeitsch. anorg. Chem.*, 1913, **84**, 277) has shown that when nitric acid is used, the arsenic must be present in the arsenic condition or notable amounts of it will be deposited with the copper. The authors confirm this and also show that accurate results can only be obtained when the current density, temperature, and other conditions are strictly limited. In ammoniacal solution accurate results may be obtained with comparatively high current densities, and no sensible error is introduced by prolonging the operation long after all the copper has been deposited, in marked contrast to the behaviour of nitric acid solutions. The arsenic must be present in the arsenic condition, arsenious arsenic giving rise to even greater errors in ammoniacal than in nitric acid solution.

Copper arsenic alloys are dissolved in nitric acid, the solution is evaporated, and the residue, together with 2·5 grms. ammonium nitrate, is dissolved in 125 c.c. of 5 per cent. ammonia. The copper is deposited from this electrolyte by a current of 5 amps. (current density, 7·5 amps./dm.^{2}) at a temperature of 90° C. The author's use Fischer's (*Electroanalyt. Schnellmethoden*, p. 78) concentric wire gauze electrodes and glass agitator, the latter being rotated at 500 to 550 revs. per minute. Under these conditions 0·3 grm. copper can be deposited in less than fifteen minutes, and the deposits are free from arsenic even when 0·2 grm. is present in the electrolyte.

G. C. J.

Separation of the Copper and Tin Groups. L. J. Curtman and J. K. Marcus. (*J. Amer. Chem. Soc.*, 1914, **36**, 1093-1103.)—As a means of overcoming the objectionable features of the ammonium polysulphide method for the separation of the copper and tin groups, the authors have investigated the sodium carbonate, sodium hydroxide, and potassium hydroxide methods taken alone or in conjunction with hydrogen peroxide. The chief features of the method recommended are embodied in the following scheme : (1) The preliminary precipitation of the alkaline earth metals, together with some of the lead before precipitation of Group 2 with hydrogen sulphide. This is effected by neutralising about 30 c.c. of the filtrate from Group 1 with ammonia, reacidifying with 5 c.c. of hydrochloric acid (1·19), adding 8 c.c. of 20 per cent. sulphuric acid, followed by boiling. The solution is then cooled and an equal volume of 95 per cent. alcohol added. (2) Treatment of the filtrate from the above with sulphur dioxide before passing in sulphuretted hydrogen in order to oxidise stannous tin and to reduce pentavalent arsenic. (3) The complete separation of the sulphides of the two divisions of Group 2 by means of potassium hydroxide. The results of a number of test analyses are given which show the method to be trustworthy. H. F. E. H.

Estimation of the Dust Fall in the Neighbourhood of Cement Plants. J. P. Mitchell. (*J. Ind. and Eng. Chem.*, 1914, 6, 454-459.)—Several glass plates (ten or more) about 5 in. by 7 in. are coated with vaseline, and exposed on a platform on the top of a pole 25 to 30 feet high, in a place away from a dusty road. After an exposure of three to five days, the plates are washed with petroleum spirit on to a filter, and the adherent dust ignited and weighed. An estimation of the calcium oxide and the silica shows approximately the proportions of "cement dust" and "field dust" in the residue, and the whole of the results are calculated into pounds per acre per day. The following results are typical of those obtained in the vicinity of a cement works. In each case ten plates were exposed.

Distance from Works (Miles).	Time of Exposure (Days).	Weight of Dust (Grms.).	Pounds per Acre per Day.	SiO_2, per Cent.	CaO, per Cent.	Calculated Cement Dust, per Cent.	Calculated Pounds per Acre per Day.	
							Cement Dust.	Field Dust.
1·2	3·69	1·015	10·9	17·92	47·95	95·5	10·4	0·5
1·2	8·14	2·079	10·1	17·29	47·39	94·3	9·5	0·6
1·7	3·84	0·563	5·8	19·59	43·19	85·2	4·9	0·9
2·2	3·77	0·061	0·7	34·86	31·09	59·1	0·4	0·3
2·8	3·88	0·634	6·5	18·52	45·17	89·5	5·8	0·7

The effect of a high wind upon the results is illustrated by the following figures, which show that the cement dust kept fairly constant whilst the amount of the field dust was increased greatly above the normal dust fall.

Conditions.	Time of Exposure (Days).	Weight of Dust (Grms.).	Pounds per Acre per Day.	SiO_2, per Cent.	CaO, per Cent.	Calculated Cement Dust, per Cent.	Calculated Pounds per Acre per Day.	
							Cement Dust.	Field Dust.
Normal	2·95	0·5125	6·8	29·9	33·7	65·0	4·4	2·4
Windy	2·95	0·5236	11·7	45·0	19·2	36·8	4·3	7·4

C. A. M.

Destruction of Organic Matter in Bog Iron Ore. L. Brandt. (*Stahl. u. Eisen*, 1914, **34**, 630-631; through *Chem. Zentralbl.*, 1914, **1**, 2069.)—The finely powdered ore is heated in a Kjeldahl flask with sulphuric acid and an oxidising agent (potassium permanganate, ammonium persulphate, or precipitated hydrated manganese peroxide) until the organic matter is destroyed. Manganese peroxide effects the oxidation more rapidly and less violently than potassium permanganate. After destruction of all organic matter, the solution is cooled, and treated with concentrated hydrochloric acid, and the iron reduced and titrated, preferably with dichromate solution, with diphenylcarbohydrazide as indicator. C. A. M.

Determination of Carbon in Steel and Iron by the Barium Carbonate Titration Method. J. R. Cain. (*J. Ind. and Eng. Chem.*, 1914, **6**, 465-468.) This comparatively little-used method has been the subject of a critical investigation in the laboratory of the United States Bureau of Standards, because, if sufficiently exact, its advantages, as compared with the use of weighed potash bulbs, are obvious, whilst it is not subject to error from access of sulphur trioxide, as in the gravimetric barium carbonate method, which has, nevertheless, found extended application. The results show that, with suitable precautions, the method is as accurate as the gravimetric methods.

The combustion is conducted in the usual manner, the carbon dioxide being absorbed by half-saturated barium hydroxide solution contained in a Meyer bulb tube, which is attached directly to the combustion-tube. At the end of the combustion, the barium carbonate is filtered off, washed free from barium hydroxide, dissolved in a measured volume of $\frac{N}{10}$ hydrochloric acid, and the excess of the latter determined by titration with $\frac{N}{10}$ sodium hydroxide. The filtration is best accomplished through about 3 cms. of quartz sand (10-30 mesh) supported on asbestos and a perforated plate contained in a carbon tube. Apparatus is figured for facilitating the filtration and washing without access of carbon dioxide. About 150 c.c. of wash-water suffices, and this quantity of pure water only dissolves 0·3 mgrm. of barium carbonate. Since the first wash-waters contain enough barium hydroxide to depress the solubility of the carbonate, the error is less than this, and with the quantities usually employed is negligible. After a little practice, a precipitate can be filtered and prepared for titration in five minutes.

There is, of course, a limit of gas speed beyond which a half-saturated solution of barium hydroxide contained in a seven-bulb Meyer tube will not remove carbon dioxide quantitatively, but the safe maximum speed can readily be established by the operator, and is the only point calling for special attention. G. C. J.

Volumetric Estimation of Lead. A. Alder and M. F. Coolbaugh. (*J. Ind. and Eng. Chem.*, 1914, **6**, 298-300.)—In spite of its apparent complexity, the following method is said to make it possible to estimate lead in an ore in less than one hour, and to be available in presence of silver, bismuth, calcium, and barium, as well as other elements less likely to interfere. It depends on separation of the lead as iodate, and titration of the latter with thiocyanate, the end-point being marked by the appearance of free iodine. The ore (0·5 grm.) is decomposed with nitric acid, with the addition of a little hydrochloric acid if necessary. Sulphuric acid (2 c.c.) is added, and the mixture evaporated until dense fumes arise. The cooled residue is treated with 100 c.c. water, the mixture boiled, alcohol (10 to 15 c.c.) is added, and the whole cooled. The supernatant liquor is decanted through a filter, and the residue and paper are washed with 10 per cent. alcohol containing 1 per cent. of sulphuric acid, and finally with 10 per cent. alcohol alone. The lead sulphate is next dissolved in 10 to 15 c.c. of a 10 per cent. solution of sodium hydroxide, the residue and filter being washed in succession with hot water, hot water slightly acidified with nitric acid, and water rendered slightly alkaline with sodium hydroxide. This treatment is necessary to insure complete solution of the lead, the use of alkaline acetate as solvent being excluded, as lead cannot be precipitated quantitatively as iodate from a solution so obtained. In presence of silver or bismuth the above treatment requires modification as the lead sulphate precipitate may retain appreciable amounts of these elements, which are transformed into hydroxides by treatment with sodium hydroxide, the hydroxides subsequently dissolving in the nitric acid used for washing. In such cases the precipitate and paper, after washing with hot water, are treated with hot water acidified with nitric acid, and the resulting filtrate is made strongly alkaline with sodium hydroxide, and refiltered through the same paper. To the combined filtrates phenolphthaleïn is added, and sufficient potassium iodate to precipitate all the lead, avoiding unnecessary excess. The mixture is heated nearly to boiling, and dilute nitric acid (sp. gr. 1·2) is added drop by drop until the pink colour is just discharged. About 1 c.c. more acid is added, the solution heated to boiling, then cooled slightly, mixed with 15 to 20 c.c. of alcohol, and finally cooled in running water. The supernatant liquor is decanted through a filter, and the precipitate is washed by decantation with 25 per cent. alcohol until iodate can no longer be detected in the filtrate, 50 c.c. of washing liquor being sufficient if no great excess of potassium iodate is used. The washed precipitate, together with the paper, is transferred to a stoppered flask, being washed in with 50 c.c. of cold dilute (2 : 5) hydrochloric acid. About 5 c.c. of chloroform are added, and the mixture titrated with ammonium thiocyanate until the chloroform acquires a violet tint not destroyed by shaking. The end-point is sharp if enough acid is present. If, after the titration has been started, it is noticed that some of the iodate is not in solution, it is best to add more dilute acid, and when much lead is present it is always advisable to add 10 to 15 c.c. of dilute (1 : 1) acid towards the end of the titration; but stronger acid must never be used, nor must the acid used for solution of the iodate be hot. The thiocyanate solution may be standardised against pure lead sulphate or metallic lead, or its lead titre may be calculated from the silver titre, when this is known, by multiplication by 1·440. The extreme error of the method need never exceed 1 in 200 if quantities are so chosen that the volume of thiocyanate finally required is not too small. A convenient strength for the thiocyanate solution is 2·5 grms. per 1,000 c.c.; 1 c.c. of such a solution is approximately equivalent to 0·005 grm. lead. G. C. J.

Sensitiveness of Various Reagents for Lead. E. Eegriwe. (*Zeitsch. anal. Chem.*, 1914, **53**, 420-426.)—Statements in the literature as to the sensitiveness of various reagents for lead being conflicting, experiments were conducted with a number of such reagents under conditions as nearly as possible identical. The experiments were made with 5 c.c. quantities of lead solution contained in test-tubes, maintained at 18° C., and examined five minutes after addition of the reagent. Dispensing with the use of control solutions, potassium dichromate was found to be the most sensitive reagent; it will show 1 part of lead in 2 millions. Used in the same way, sodium sulphide fails to detect 1 part in 1 million, but with the help of a control solution this reagent will show 1 part in 3 millions. Ammonium molybdate will show 1 part in 800,000. Potassium dichromate, ammonium molybdate, and some other reagents are most effective when only small quantities are used, such as 0·05 c.c. of $\frac{N}{1}$ solution to 5 c.c. G. C. J.

Analysis and Composition of Red Lead. A. P. West. (*Philippine J. of Science*, 1914, **8**A, 429-437.)—The sum of insoluble matter (when present), of free litharge, estimated by extraction with a solution of lead nitrate, and of Pb_3O_4 calculated from the weight of lead dioxide, separated in the usual manner, never adds up to 100 per cent. It is shown that this is due to the fact that red lead, freed from litharge, does not consist of Pb_3O_4, but has a composition which corresponds to a mixture of Pb_3O_4 and Pb_4O_5. A genuine product, consisting solely of oxides of lead, may be analysed with sufficient exactness by determining free litharge and lead dioxide in the usual manner, and calculating the percentage of combined lead monoxide by subtracting from 100 the sum of the percentages of litharge and lead dioxide. From the ratio of lead dioxide to combined lead monoxide, the percentages of apparent Pb_3O_4 and Pb_4O_5 can be calculated.

A method of more general application consists in estimating insoluble matter, free litharge, lead dioxide, and total lead monoxide in the filtrate from the latter. This last estimation is seldom invalidated by the presence of soluble lead salts. The difference between total lead monoxide and free litharge gives combined monoxide, and from this and the lead dioxide the percentages of Pb_3O_4 and Pb_4O_5 can be calculated. A table is given to facilitate the calculation. Expressed in this way, the results of a number of analyses show that genuine red lead may contain from 5 to 20 per cent. of Pb_4O_5 as well as Pb_3O_4 (50 to 80 per cent.) and free litharge (5 to 40 per cent.), but that Pb_4O_5, as well as the other oxides, is apparently always present.

Insoluble matter is most conveniently estimated by Baucher's method (*Petit Monit. Pharm.*, **47**, 2651), 2 grms. of the sample being boiled with 100 c.c. of 10 per cent. nitric acid and the lead dioxide reduced and brought into solution by adding 15 c.c. of sugar solution (1 : 3) after a few minutes. The insoluble matter is then filtered off on a Gooch crucible.

Free litharge is best extracted with lead nitrate (not acetate). Half a grm. of the sample is digested on the water-bath for an hour with 100 c.c. of 10 per cent. lead nitrate; the mixture is then boiled for ten minutes, diluted with 100 c.c. hot water to dissolve basic salts, and filtered through a Gooch crucible. The washed residue is weighed and litharge estimated by difference. (*Cf.* Lowe, *Ding. polyt. J.*, 1889, **271**, 472.)

Lead dioxide is estimated by the method of Lux, digesting 0·5 grm. on the water-bath for thirty minutes with 100 c.c. of 10 per cent. nitric acid, filtering through a Gooch crucible, washing and weighing the residue, and correcting for any insoluble matter. In the filtrate from the lead dioxide, total lead monoxide is estimated in any convenient manner. G. C. J.

Estimation of Nickel by Means of Dimethylglyoxime. O. Brunck. (*Zeitsch. angew. Chem.*, 1914, **27**, 315.)—A survey of and reply to the criticism to which this method has been subjected. It is admitted that a distinct precipitate of nickel dimethylglyoxime occasionally forms in the filtrate when the principal precipitate is filtered off at once without delay, as originally recommended by the author (*Zeitsch. angew. Chem.*, 1907, **20**, 1844); but it is shown that the nickel thus lost is far smaller in amount than the voluminous nature of the precipitate might suggest, in no case reaching 0·2 mgrm., and generally being much less. For the most exact work, a period of an hour may be allowed before filtration, but in most cases the slightly increased accuracy thus obtained does not justify the loss of time involved. A great excess of ammonia slightly increases the solubility of the precipitate, as does a high concentration of alcohol, such as may result when a large quantity of the alcoholic solution of the reagent is added to a comparatively concentrated aqueous solution of nickel. Ammonium salts, even in large quantity, are without effect.

In contradiction to Iwanicki (ANALYST, 1909, **34**, 74), the author finds that the precipitate can be ignited to oxide without loss, as described by Wdowiscewski (*Stahl. u. Eisen*, 1908, **28**, 960) and others, but he prefers to collect in a Neubauer crucible, dry at 110° to 120° C. for forty-five minutes only, and weigh the undecomposed compound, as this weighs four times as much as the equivalent amount of nickel oxide, and makes it possible to work with small quantities. The use of large quantities of nickel steel involves an ether separation of the greater part of the iron, since the use of enough tartaric acid to hold all the iron in solution would interfere with the subsequent precipitation of the nickel. Since Neubauer crucibles are costly, the author has obtained from W. C. Heraeus, in Hanau, a number of porcelain Gooch crucibles with a mat of platinum black. These serve well. They tend to adsorb traces of silica in time and then filter badly, but are readily restored to their former condition by treatment with a little dilute hydrofluoric acid.

König (ANALYST, 1913, **38**, 527) has stated that nickel cannot be separated from cobalt by means of dimethylglyoxime. This is only true when ferric iron is simultaneously present. In these circumstances the precipitate is contaminated by a compound of cobalt and iron with dimethylglyoxime. When the precipitation is made in acetic acid solution, for which purpose the iron must be in the ferrous condition even in absence of cobalt, the results are exact. Exact results may also be obtained in ammoniacal solution, if this is reduced, whilst still concentrated, with a saturated solution of sulphurous acid, of which an excess of 5 to 10 c.c. is then added to prevent reoxidation. The solution is then diluted with hot water and the nickel precipitated with dimethylglyoxime. Five times the weight of the nickel and cobalt present is a suitable quantity to add. (*Cf.* Rhead, ANALYST, 1910, **35**, 97.) G. C. J.

Estimation of Minute Amounts of Sulphur Dioxide in Air. A. Seidell and P. W. Meserve. (*J. Ind. and Eng. Chem.*, 1914, **6**, 298-301.)—The problem to be solved was the approximate estimation of sulphur dioxide in such concentrations as may occur in railway tunnels, that is to say, of the order of 10 parts per million. Methods based on the aspiration of a large amount of air through a small quantity of liquid were excluded, as it was required to estimate the concentration of sulphur dioxide at any particular instant. The following method is recommended: A large (2,500 c.c.), dry, evacuated bottle is opened at the place where the air is to be tested and then, with the least possible delay, 5 c.c. of 0·1 per cent. starch solution are introduced, caused to wet the whole internal surface of the bottle, and titrated with $\frac{N}{1000}$ iodine solution. If the bottles and air are dry, and the analysis is completed within half an hour, the results multiplied by 1·3, to correct for unavoidable oxidation by air, are sufficiently exact. The use of an excess of iodine and back titration with thiosulphate gives higher but less concordant results, the end-point at such dilutions being too indefinite. If the bottles are wet or the air supersaturated with moisture, the influence of even half an hour's delay in completing the analysis may be serious.

G. C. J.

Sources of Error in the Analysis of "Thomas" Meal. M. Popp. (*Chem. Zeit.*, 1914, **38**, 741-742.)—Concordant results may be obtained by observing numerous precautions, of which the following are the most important. To prevent caking, it is advisable, before adding the citric acid solution, to moisten the meal with 5 c.c. of alcohol, and to start the shaking without loss of time (*cf.* ANALYST, 1907, **32**, 241). In filtering the extract, the whole of the liquid should be poured on to a large filter, so as to prevent further action of the citric acid solution upon the residue in the flask. In the case of certain meals phosphoric acid may separate simultaneously with a deposit of calcium citrate. For the precipitation the 50 c.c. of the filtrate are treated successively with 25 c.c. of the iron citrate solution, 1 c.c. of 3 per cent. hydrogen peroxide solution, and 25 c.c. of magnesia mixture. The addition of more than 3 c.c. of hydrogen peroxide solution causes the results to be too high. After shaking, the precipitate may be left for several days without affecting the results, provided the flask is kept cool. The ignition must be done under constant conditions, and for only a short time. Loss by volatilisation may be prevented by heating the precipitate in a platinum crucible on an aluminium plate, covering it with a piece of graphite, and then igniting it for exactly five minutes, supported in a quartz cylinder attached to the top of a Teclu burner.

C. A. M.

Estimation of Citric Acid Soluble Phosphoric Acid in Basic Slag. F. Haussding. (*Landw. Jahrb.*, 1913, **45**, 119-126; through *Chem. Zentralbl.*, 1914, I., 2016.)—The method proposed by Popp, in which iron citrate is employed (ANALYST, 1913, **38**, 526), yields results which agree with those obtained when the silica is removed by treatment with hydrochloric acid; Lorenz's more accurate method, however, gives results which are lower than those obtained by either of the above-mentioned methods.

W. P. S.

Estimation of Citric Acid Soluble Phosphoric Acid in Basic Slag by the Citrate Method and Lorenz's Method. H. Neubauer. (*Landw. Versuch. Stat.*, 1914, **82**, 465-475; through *Chem. Zentralbl.*, 1914, I., 2015-2016.)—Lorenz's method yields accurate results, and the presence of citric acid in the solution of the slag does not interfere with this method. The citrate method always yields results which are too high even when iron citrate solution is added to prevent the precipitation of silica; the error is due, principally, to the precipitation of a small quantity of tricalcium phosphate, and is more pronounced in the case of basic slag than with superphosphates (soluble phosphoric acid estimation), owing to the larger proportion of calcium salts in the former. W. P. S.

Estimation of Thorium in Monazite Sand. R. J. Carney and E. D. Campbell. (*J. Amer. Chem. Soc.*, 1914, **36**, 1134-1143.)—The method depends on the insolubility of thorium pyrophosphate in dilute acids, the pyrophosphates of the other rare earths, including cerous (but not ceric) cerium being soluble. Like other phosphates, the precipitate renders the combustion of the filter ash difficult, and, as an oxalate precipitation is necessary at some point in the analysis to eliminate zirconium, the pyrophosphate of which, like that of thorium, is insoluble in dilute acids, this is made the last step and the thorium finally separated as oxalate and weighed as oxide.

Fifty grms. of the sand are heated in an iron dish with 100 c.c. of sulphuric acid at about 250° C. until decomposition is complete (five to eight hours), and the cooled mixture is poured into 400 c.c. of water. When the sulphates have all dissolved, the solution is filtered and made up to 1,000 c.c., of which 50 c.c. are transferred to a large beaker, mixed with 5 c.c. of hydrochloric acid and diluted to about 450 c.c. About 15 c.c. of a 5 per cent. solution of sodium pyrophosphate are added slowly with constant stirring and the mixture is heated to boiling, boiled for five minutes, allowed to stand for five minutes, and then filtered. The precipitate, which contains about 15 mgrms. of other oxides besides thoria and zirconia, is washed several times with hot water acidified with hydrochloric acid, and is then transferred with the paper to a Kjeldahl flask and digested with 15 c.c. sulphuric acid and a few crystals of ammonium perchlorate to destroy the paper. The mixture is heated, and when it changes to a clear brown liquid, more perchlorate is added and the heating continued until all organic matter is oxidised, but the sulphate obtained at this stage is always slightly yellow. About ten minutes and 1·5 grms. of perchlorate suffice to destroy a 15 cm. filter. The contents of the flask are cautiously diluted with 100 c.c. of water and, when the sulphates have dissolved, are poured into a solution of 30 grms. sodium hydroxide in about 125 c.c. of water. The mixture is boiled for several minutes and the hydroxides are then filtered off on a hardened paper, using suction, and are washed with hot water. The paper and precipitate are returned to the beaker, treated with 10 c.c. of hydrochloric acid (sp. gr. 1·19), and, after a short time, 150 c.c. of water are added and the solution boiled and freed from paper by filtration. To the filtrate, 3 c.c. of a saturated solution of sulphurous acid are added to reduce traces of ceric salts, the mixture is heated to boiling and the thorium freed from the last traces of other earths, except zirconium, by reprecipitation with sodium pyrophosphate. The precipitate is transformed into sulphate and hydroxide as before, the sulphate this time being colourless. The hydroxide is dissolved in 5 c.c. of hydrochloric acid, the solution freed from paper as before by filtration, and the thorium precipitated from the filtrate by a solution of 2 grms. of oxalic acid. The mixture is diluted to 450 c.c. and left in a warm place overnight. The oxalate is then filtered off, washed with water faintly acidified with hydrochloric acid, and ignited to oxide.

Excellent results were obtained with a number of artificial mixtures of known composition, but in these mixtures the other earths did not preponderate as they do in monazite. On monazite containing about 5·5 per cent. of thoria the results obtained by the new method do not, on the average, differ from those yielded by the iodate method by more than 1 in 500, and when a number of determinations are made on the same sample the extreme variation from the mean is only 1 in 150.

G. C. J.

Gravimetric Estimation of Tellurium. A. Gutbier and J. Huber. (*Zeitsch. anal. Chem.*, 1914, **53**, 430-433.)—It is pointed out that Rosenheim and Weinheber's criticism (ANALYST, 1911, **36**, 179) of Gutbier's method is based on a misreading of that method, as Gutbier did not recommend reduction in alkaline solution. A better method than Gutbier's original method, however, is that of Lenher and Homberger (ANALYST, 1908, **33**, 249), in the author's opinion the best method yet available for the gravimetric estimation of tellurium. Test numbers are given which show that the maximum error of the method is 0·4 mgrm., which corresponds to a very small percentage error when the final weighing is of 0·2 grm. or more.

G. C. J.

Separation and Estimation of Tungstic Acid. A. Gutbier and G. L. Weise. (*Zeitsch. anal. Chem.*, 1914, **53**, 426-430.)—When a solution of alkali tungstate is acidified with acetic acid and treated hot with nitron, the tungsten is completely precipitated The solution of alkali tungstate, containing 0·1 to 0·2 grm. tungstic acid and as little free alkali as possible, is diluted to about 150 c.c. with boiling water, and acidified with acetic acid. Whilst stirring continually, an excess of nitron acetate (Busch, *Ber.*, 1905, **38**, 861) is added slowly to the boiling solution, after which the boiling must cease, or filtration will be difficult. The precipitate is filtered off, washed with 100 c.c. of 0·4 per cent. nitron acetate, transferred with the paper—either wet or dry—to a crucible, cautiously decomposed, and finally ignited and weighed as WO_3. Water cannot be used to wash the precipitate, and the direction to have no more alkali salts present than need be is dictated by the fact that the removal of large quantities of alkali salts from the filter would necessitate the use of more than 100 c.c. of the somewhat expensive reagent.

G. C. J.

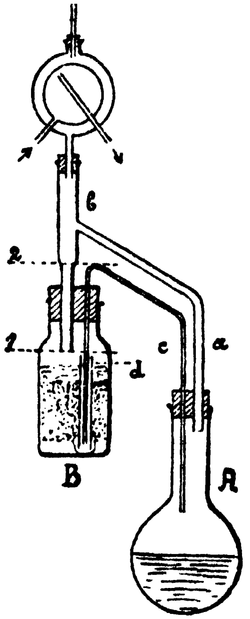
Estimation of Hardness in Water. L. W. Winkler. (*Zeitsch. anal. Chem.*, 1914, **53**, 409-415.)—Total hardness is best determined by Blacher's method (ANALYST, 1913, **38**, 127), but 1 drop of methyl orange solution (1 : 1000) is used as indicator, instead of dimethylaminobenzene, in the preliminary neutralisation, and it is unnecessary to remove carbon dioxide quantitatively from the solution. Large quantities of carbon dioxide must be removed by blowing air through the solution or allowing to stand for twenty-four hours, but a small quantity (1 c.c. per 100 c.c.) makes the end-point of the ultimate titration with potassium palmitate more sharp. The methyl orange is destroyed by the addition of 1 drop of dilute bromine water before adding 1 c.c. of 0·5 per cent. phenolphthaleïn solution. $\frac{N}{10}$ sodium hydroxide is next added until the solution is strongly pink, and then $\frac{N}{10}$ hydrochloric acid until the pink colour is just discharged. An additional 0·1 c.c. of $\frac{N}{10}$ hydrochloric acid is added, and potassium palmitate then added from a burette until a distinct pink tint reappears. In presence of a trace of carbon dioxide the precipitate of alkaline earth palmitates appears snow-white almost until the end-point is reached, but the best end-point to work to is a very distinct red rather than the first appearance of pink, 0·3 c.c. being deducted from the reading. The palmitate solution must be prepared from palmitic acid free from stearic acid, 25·6 grms. being introduced into a flask together with 500 c.c. alcohol, 300 c.c. water, and 0·1 grm. phenolphthaleïn, and saponified by addition of alcoholic potash to the hot solution, which should finally be just distinctly alkaline. The solution is made up to 1,000 c.c. when cold, and standardised by taking 50 c.c. of saturated lime-water, neutralising this with accurately standardised $\frac{N}{10}$ hydrochloric acid, using methyl orange as indicator, diluting to 100 c.c., and then treating the diluted solution exactly as described for the estimation of hardness, including the final deduction of 0·3 c.c. The volume of palmitate solution required, thus corrected, should equal the volume of $\frac{N}{10}$ acid required to neutralise the lime-water.

For the estimation of hardness due to lime, the following improved modification of the author's method (*Zeitsch. anal. Chem.*, 1901, **40**, 82) is recommended. The solutions required are —(1) potassium oleate solution prepared by saponifying 12 grms. almond oil with 3 grms. of potash dissolved in 100 c.c. of alcohol and dilution to 1,000 c.c. with 50 per cent. alcohol; (2) alkaline tartrate solution containing 100 grms. Rochelle salt, 10 grms. sodium hydroxide, and 250 c.c. of 10 per cent. ammonia in 500 c.c. To 100 c.c. of the water to be tested, 5 c.c. of the alkaline tartrate solution are added, and then potassium oleate from a burette until, on shaking, a persistent froth is obtained. The oleate solution is standardised by taking about 10 c.c. of saturated lime-water, neutralising with accurately standardised $\frac{N}{10}$ hydrochloric acid, using methyl orange as indicator, destroying the indicator with 1 drop of dilute bromine water, diluting to 100 c.c., and titrating as described. One c.c. of the oleate solution corresponds approximately to one German degree of hardness when 100 c.c. of water are taken for titration. G. C. J.

Colorimetric Estimation of Traces of Manganese in Water. H. Lührig. (*Chem. Zeit.*, 1914, **38**, 781-783.)—One hundred c.c. of the water are treated with 3 c.c. of nitric acid (sp. gr. 1·40) and sufficient silver nitrate solution to precipitate the whole of the chlorine, a slight excess being negligible. About 3 grms. of ammonium persulphate (in solid form or solution) are added, and the mixture boiled for five minutes, and then rapidly cooled. In the presence of manganese salts the coloration of permanganic acid will appear, and may be matched with water to which definite quantities of manganese have been added and which has been treated in the same way. If too little silver nitrate has been added, the colour has a reddish tint, whilst in the presence of a large excess of silver nitrate the tint is bluish. It is therefore advisable to add sufficient sodium chloride to the standard liquid to correspond to the chlorine in the water. The sharpest comparisons are possible by the use of 100 c.c. of water containing from 0·3 to 0·6 mgrm. of manganese per 1,000 c.c., but it is possible to distinguish between the colorations given by 0·1 and 0·2 mgrm. per litre. The method is applicable to waters containing iron salts, but in such cases it is advisable first to boil the water for a few minutes with 1 grm. of ammonium persulphate, then to add the nitric acid and the calculated quantity of silver nitrate, and finally to complete the oxidation with the remainder of the ammonium persulphate. C. A. M.

APPARATUS, ETC.

Simple Extraction Apparatus. M. Freund. (*Chem. Zeit.*, 1914, **38**, 802.)—The material to be extracted is placed between layers of cotton-wool in the flask *B*, which is connected, as shown in the diagram, with the tared flask *A*. The evaporated solvent passes through *a*, is condensed, and after passing through the material is siphoned back into *A*, without touching the cork of *B*. C. A. M.



Thermo-Electric Method for the Determination of the Purity of Platinum Ware. G. K. Burgess and P. D. Sale. (*J. Ind. and Eng. Chem.*, 1914, **6**, 452-454.)—The method depends on the fact that the thermo-electromotive force of platinum against many of its alloys has been determined with considerable exactness. Unlike the very exact method depending on measurement of temperature coefficient of electrical resistance, which is only applicable to wires, the new method can be applied to platinum vessels of any form, and without defacing them. To the crucible or other vessel, at two points as far apart as possible, two pure platinum wires (Heraens' normal thermo-element wire, 0·1 to 0·2 mm.) are arc-soldered. The platinum wires are connected to an ordinary pyrometer, galvanometer, or millivoltmeter, and another wire of platinum-rhodium (9 : 1) is also arc-soldered to the crucible very close to (0·5 to 1 mm. distant), but not touching, one of the other junctions, which is intended to be the hot junction. By means of a commutator, the platinum-rhodium wire may be connected to one terminal of the galvanometer instead of the platinum wire from the cold junction. The cold junction is suitably screened and kept cool by an air blast, whilst the small area including the other two junctions is raised to a high temperature by means of a blowpipe flame. With the commutator in one position this temperature is recorded, whilst in the other the reading is a measure of the electromotive force of the thermocouple, consisting of the crucible and the pure platinum. Since iridium is the commonest impurity of platinum, the total impurities are estimated in terms of iridium by reference to isothermal curves connecting the E.M.F. of a platinum-platiniridium couple with the percentage of iridium in the alloy. Three such curves (900 to 1100° C.) are given in the paper. The method does not distinguish the various possible impurities, but all the other platinum metals, when alloyed with platinum, give at high temperatures an E.M.F. of the same sign against pure platinum, the anomalous behaviour of alloys of high palladium content (>90 per cent.) not constituting an exception of any importance in this connection. There is therefore no possibility of one impurity balancing the effect of another, and, failing better methods, this one, which requires little time, has been found of service in the U.S. Bureau of Standards, where it was worked out. G. C. J.

Resistance of Platinum Vessels to Hot Nitric Acid. G. P. Baxter and F. L. Grover. (*J. Amer. Chem. Soc.*, 1914, **36**, 1089-1091.)—Jannek and Meyer (*Zeitsch. anorg. Chem.*, 1913, **83**, 51) have stated that 10 c.c. of halogen-free nitric acid distilled through a platinum condenser yielded 0·00228 grm. of non-volatile material upon evaporation in a platinum crucible. The authors find that this surprisingly large residue is to be attributed either to the use of vessels of impure platinum, to inefficient cleaning of the platinum vessels, or to the presence of traces of hydrochloric acid in the nitric acid employed. With platinum vessels which have not been subjected to drastic cleaning processes, the danger of solution of surface impurities is serious. New platinum should be thoroughly cleaned either by fusion with acid potassium sulphate or by sublimation of ammonium chloride from the vessel as recommended by Stas. H. F. E. H.

Silver-Plated Copper Gauze Electrode in Zinc Determination. O. L. Barnebey. (*J. Amer. Chem. Soc.*, 1914, **36**, 1144.)—A silver-plated copper gauze electrode can be made cheaply and is a good substitute for platinum in the electrolytic deposition of zinc. Copper gauze is cut to a convenient size, the ends brought together to form a cylinder, and all the edges folded down. The ends are joined by interlocking folds. The electrode is supported by means of a heavy copper wire which encircles the cylinder within the upper fold and is bent at right angles for connection to the support. The folds are then sewn together by means of a copper wire. The cylinder and rod are immersed in a dilute cyanide silver bath, and coated with silver by electrolysis, after which the unplated portion of the rod is cut off. A platinum wire is used for the anode connection. After each analysis the zinc can be removed conveniently by means of dilute hydrochloric acid. G. C. J.

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REVIEW.

PRINCIPES D'ANALYSE ET DE SYNTHÈSE EN CHIMIE ORGANIQUE. M. HANRIOT, P. CARRÉ, A. SEYEWETZ, E. CHARABOT, and A. HÉBERT. 795 pp. Librairie Polytechnique. 1914. Ch. Béranger, Paris et Liège. Price 30 f.

This work forms the fifth volume of the "Encyclopédie de Science Chimique Appliquée," published under the direction of Professor Chabrié. The book is divided into five parts, dealing respectively with the general principles of organic analysis, pharmaceutical products, methods of synthesis of colouring matters, organic perfumes, and a general study of saponification. Each of the authors mentioned above as collaborating in the work has been responsible for a section, the order in which authors and sections are given being the same.

The general impression gained by the reviewer is that an attempt has been made to cover too much ground. This is very noticeable in the first section of 132 pages. The first chapter of this section deals with ultimate analysis, determination of molecular weights and molecular formulæ, and constitutions of organic compounds. The twenty-nine pages of Chapter II. are concerned with proximate analysis, the separation of organic substances, and determination of physical constants; although condensed, this chapter contains much good material. But the fifty-eight pages of Chapter III. do not suffice for the "Description des Espèces" which is attempted. Common hydrocarbons, organic acids, aldehydes, ketones, quinones, sugars, amino-acids, a few alkaloids, and a good many dyestuffs are mentioned in this chapter, and, needless to say, lists of substances and tests for their identification are necessarily incomplete.

Professor Carré's section on pharmaceutical products is of very considerable interest, the first forty pages of this section being taken up with an account of the relationship of physiological properties to constitution, followed by an account of the chief chemical products employed in medicine. This section occupies 215 pages, the next section, relating to dyestuffs, being even longer—nearly 300 pages in fact. M. Seyewetz has given a good general account of the organic dyestuffs, introducing the subject with general considerations as to the cause of selective absorption, and proceeding afterwards to a description of the various groups of dyestuffs. Anyone reading this section would get a good general idea of the state of dyestuff chemistry, but 300 pages do not suffice to go into much detail in so extensive a branch.

The two last sections are shorter, perfumes occupying about seventy pages, whilst saponification is dealt with in fifty pages. The work concludes with a table of contents occupying ten pages. There is, however, no index—a most regrettable feature in a work of this character. As usual with French scientific works, this defect is remedied to a certain extent by the completeness of the table of contents, whilst printing and illustrations are excellent. J. T. HEWITT.

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INSTITUTE OF CHEMISTRY.

PASS·LIST: JUNE–JULY (1914) EXAMINATIONS.

The results of the Examinations of the Institute of Chemistry recently held in London, Dublin, and Glasgow have now been published. Of twenty-three candidates who presented themselves for the Intermediate Examination, eleven passed: A. J. Boyd, R. G. Browning, R. C. Denington, A. O. Jones, J. King, E. Leitch, B.Sc. (Glas.), G. W. Moore, P. D. Oakley, P. W. Rait, E. L. J. Stockdale, B.Sc. (Lond.), and H. M. Webb. Of twenty-eight candidates who presented themselves for the Final (A.I.C.) Examination, thirteen passed—In the Branch of Mineral Chemistry: H. Trickett and H. Vernon, B.Sc. (Lond.); in the Branch of Organic Chemistry: J. G. Duncan, T. H. Durrans, B.Sc. (Lond.), S. H. Groenewoud, L. A. Jordan, B.Sc., A.R.C.S. (Lond.), L. Orange, B.Sc. (Lond.), W. S. Ritchie, B.Sc. (Lond.), F. W. Snelgrove, B.Sc. (Lond.), J. W. Tait, M.A. (Edin.), and F. D. White; in the Branch of the Chemistry (and Microscopy) of Food and Drugs, Fertilisers and Feeding Stuffs, Soils, and Water: J. H. Christie, B.Sc. (Lond.), and C. G. Collins. Two candidates examined at Johannesburg have also satisfied the Board: In the Branch of Metallurgical Chemistry, D. Millin, B.A. (Cape), and in the Branch of Organic Chemistry, A. Kloot, B.Sc. (Lond.).

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THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

THE PROPERTIES OF SOME CHLORHYDROCARBONS AND THEIR USES IN CHEMICAL ANALYSIS.

By L. GOWING-SCOPES.

PART II.—THE ESTIMATION OF THE CHLORETHANES.

INVESTIGATION CARRIED OUT UNDER THE SOCIETY'S ANALYTICAL INVESTIGATION SCHEME.

(*Read at the Meeting, June* 3, 1914.)

IN an earlier paper on this subject (ANALYST, 1914, 4) I stated that " An interesting reaction apparently takes place between pentachlorethane and phenylhydrazine at ordinary temperatures when mixed and allowed to stand. A copious evolution of gas takes place, and a solid compound is formed which, when washed with petroleum ether (in which it is insoluble), to free it from excess of phenylhydrazine or pentachlorethane, is of a dark golden colour."

This reaction has now been further studied, and the compound is found to be phenylhydrazine hydrochloride. Nitrogen and benzene were also formed. The reaction was also found to be given by hexachlorethane, and in a lesser degree by tetrachlorethane. The chlorethylenes gave a feeble reaction, due, no doubt, to the presence of chlorethanes as impurities.

The reaction may be expressed as follows :

$$C_2H_2Cl_4 + C_6H_5.NH.NH_2 = C_2H_2Cl_2 + 2HCl + N_2 + C_6H_6$$
$$C_2HCl_5 + C_6H_5.NH.NH_2 = C_2HCl_3 + 2HCl + N_2 + C_6H_6$$
$$C_2Cl_6 + C_6H_5.NH.NH_2 = C_2Cl_4 + 2HCl + N_2 + C_6H_6.$$

Any one of the four substances formed, if estimated, would be a measure of the chlorethanes.

The estimation of chlorethylenes or benzene was at once dismissed as impracticable. The estimation of the hydrochloric acid and nitrogen was tried, but it was found that the reaction depended to a very large extent upon the conditions of the experiment, particularly the proportion of phenylhydrazine to chlorethane present, dilution, and temperature.

In the experiments where nitrogen was estimated, the substance was dissolved in xylene, and treated with phenylhydrazine in excess. The gases were first passed through a reflux condenser (to condense xylene) and then into a gas burette. Unfortunately, the gases cannot be driven out by carbon dioxide and collected in a Schiff nitrometer, as carbon dioxide reacts with phenylhydrazine. Working in this manner, there was little difficulty in obtaining good results with hexachlorethane, the reaction apparently going quite uniformly; but with the other chlorethanes, less than half the gas was evolved. The probable explanation of this is that in those compounds which contain hydrogen as well as chlorine, the phenylhydrazine acts more in the nature of an alkali, hydrochloric acid splits off and combines with the phenylhydrazine to form phenylhydrazine hydrochloride without the phenylhydrazine molecule splitting up. This appeared to be confirmed by the fact that if small quantities of pentachlorethane are added to relatively large quantities of phenylhydrazine and xylene, practically no gas is given off, even after prolonged boiling. Two reactions, therefore, seem to go on simultaneously: one where two chlorine atoms are removed from the chlorethane and the phenylhydrazine splits up, and the other where the phenylhydrazine, acting like an alkali, removes one atom of hydrogen and one of chlorine as hydrochloric acid. The conditions of the experiment determine which of these is the main reaction. A modifying effect is apparently produced by the solubility of phenylhydrazine hydrochloride in toluene, or in xylene containing phenylhydrazine.

As hexachlorethane contains no hydrogen, it would be expected that it would either give the calculated quantity of gas, or would not react at all. Experiments to test this point showed that the reaction proceeded quite normally.

The amount of hydrochloric acid formed likewise varies with the conditions of the experiment, so that the estimation of this substance is also out of the question.

When making experiments on the phenylhydrazine reaction, an alcoholic solution of an alkali was added to see if it was possible to prevent the formation of the phenylhydrazine hydrochloride. As soon as the solution was added, a precipitate of alkali chloride was formed even in the cold. This reaction proved extremely useful, and affords an easy method of estimating the chlorethanes, with the exception of hexachlorethane, even in the presence of the chlorethylenes.

The experiments were conducted in the following manner:

An approximately $\frac{N}{1}$ solution of potassium hydroxide in absolute alcohol was prepared, any potassium carbonate being removed by filtration. Into a dry 100 c.c. flask 10 c.c. of the alkali solution was introduced without wetting the neck, 20 c.c. of xylene were added, and the whole stoppered and weighed. A few drops of the chlorhydrocarbon were rapidly dropped direct into the liquid, the flask quickly restoppered and reweighed. After allowing to stand for a few minutes, water was added to dissolve the precipitated halide and the solution made acid with dilute nitric acid, the chlorine being estimated by means of silver nitrate and thiocyanate. From the results obtained it was found that under these conditions 1 molecule of hydrochloric acid splits off. That is—

1 c.c. $\frac{N}{10}$ silver nitrate = 0·0168 tetrachlorethane.
" " " = 0·0202 pentachlorethane.

The following results were obtained :

Tetrachlorethane.			Pentachlorethane.		
Taken.	Found.	Dif.	Taken.	Found.	Dif.
Grm.	Grm.	Grm.	Grm.	Grm.	Grm.
0·1515	0·1512	- 0·0003	0·1182	0·1143	- 0·0039
0·1770	0·1764	- 0·0006	0·0906	0·0879	- 0·0027

It will be noted that the results are low, and that the agreement for pentachlorethane is worse than that for tetrachlorethane. This is due to the fact that both compounds contain hexachlorethane which does not react, tetrachlorethane containing about 0·3–0·5 per cent., and pentachlorethane about 3–5 per cent.

The amount of hexachlorethane in tetrachlorethane and pentachlorethane can thus be estimated. This will be useful to workers with cellulose acetates, as hexachlorethane acts as a camphor substitute.

The following conditions were found necessary :

(1) The amount of chlorhydrocarbon taken should not be more than about 0·15 grm. (2) The solution must not be heated or allowed to stand for a great length of time. (3) The alkali solution to be made up with xylene to approximately $\frac{N}{3}$. If the chlorethane is dissolved in some solvent before being added to the alkali, less xylene will be required. Stronger alkali appears to carry the reaction farther, while if the alkali is too dilute the reaction is not complete.

It was found that this reaction did not take place with either dichlorethylene, trichlorethylene, perchlorethylene, chloroform, or carbon tetrachloride. On prolonged standing, a very small precipitate settled out. If the solution is now boiled, a heavy precipitate comes down. In the case of trichlorethylene, the sodium ethylate in the solution reacts with the formation of dichlorvinyl ether. No doubt a similar reaction takes place with the other two; whether this is quantitative, and if the precipitation of the chlorethylenes can be effected by means of alkaline mercury cyanide, is left for a further communication.

It was also found that if the solution of hexachlorethane in xylene alkali solution is boiled for some time or allowed to stand a day or two, the solution acquires a fairly deep orange colour. What this is due to has not been found out. None of the other chlorhydrocarbons give this, even on prolonged standing, so it appears that the hexachlorethane must gradually enter into combination in a different manner from the others. An investigation is in hand to see whether this will afford a colorimetric method of estimating hexachlorethane.

In conclusion, I have to thank Messrs. McLachlan and Nicholson for help with the work.

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NOTE ON THE INSOLUBLE BROMIDE VALUE OF OILS.

By JOHN A. L. SUTCLIFFE, A.I.C.

Recently (Analyst, 1914, 28) the author published a note on the above subject, in which a method of determination was suggested. Gemmell (Analyst, 1914, 297), in criticising that method, makes the following statement: "Sutcliffe himself states, also, that the factor (0·63 I.V.—78) may vary with different investigators, and, if such is the case, it would be impossible to lay down constants for the guidance of analysts as a means of detecting adulteration. Still another objection is the disregard of a constant temperature, that of tap-water, as he recommends, being liable to great seasonal variation." No statement was made in the author's paper as to the variation *with different analysts*. The effect of such a statement, in view of the opening paragraph, would be to stultify the paper. The reference to the variation of the constant (78) in place of the factor (0·63) was that variation in the *conditions* already laid down would alter the equation in the manner indicated—a very different thing.

This remark was inserted for three reasons. First, to warn any person who might wish to vary the standard conditions in any way. Second, to draw attention to the peculiar coincidence of the relationship of 0·63 I.V. to the bromine value. Third, in order that it might be referred to in a further paper. With regard to the process itself, sufficient standard data were given in the former method, and in this connection the following facts may be noted:

The temperature of cooling before and during precipitation (11° C.) is stated simply to prevent the formation of substituted bromine compounds, and is quite accurate enough for this purpose. This also applies to the direction as to standing in water overnight. Even in summer the temperature of running tap-water is sufficiently low to prevent substitution.

A direction to cool before filtration might have been inserted, but, in view of the remarks previously made as to the solvent power of uncooled ether, it was thought to be unnecessary. If the above conditions are observed, there only remains the condition during washing, and here more detail may be inserted. Before filtration the flask and contents are cooled to below 5° C., and the red ether solution is decanted through the filter. The bromides are washed with cooled ether, and whilst settling, the filter-paper is washed free from bromine with an ether jet. The washing and filtration are then carried out as before. Unless the bromides when dried are white and crumbly the test is to be rejected. It has been found that some samples of bromine give precipitates that are horny and dark on drying. As to the accuracy of the process; the precipitate, as Gemmell points out, is not the pure hexabromide, or even apparently the bromide of a mixed glyceride, but the object of my former paper was to arrive at a fairly quick process capable of giving reasonably constant results. In performing the test in conjunction with two or three other chemists, and without any *special* care being taken, the variation is generally well within 1 per cent. If care be taken duplicate experiments should be within 0·5 per cent.

In view of these figures, it appears that the above test is at least as capable of forming a basis for the formation of standards for the detection of adulteration as one in which oils of such varying iodine values as 175 and 185 give the same percentage of bromides. This would indicate that little is to be gained by the extra process of the separation of fatty acids.

This being so, it is still maintained that an accuracy of 0·5 per cent. is sufficient for an analytical test on such substances as oils, and that the separation and examination of the fatty acids are needless.

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THE FACHINI-DORTA METHOD OF [APPROXIMATELY] SEPARATING LIQUID FROM SOLID FATTY ACIDS IN OILS AND FATS.

By ARMAND DE WAELE.

An article in the *Chemical World* (1912, 1, 97) on this method was published by the authors, but the details given being somewhat incomplete may probably account for the obscurity and small popularity of a method which I have found to satisfactorily replace the older and more cumbersome lead-salt-ether method. I have used the method now for over two years in connection with researches on both raw and oxidised oils for use in linoleum manufacture, and can amply testify both to its simplicity and greater ease of manipulation over the older method.

It will be noted that all the methods hitherto known for effecting separation of saturated from unsaturated fatty acids are, at the best, only approximate in accuracy, the solubility of the potash soaps or lead soaps in the various media employed being proportional to the degree of unsaturation, hence oleic acid is apt to behave as intermediate in solubility between the saturated acids and those of the greatest unsaturation. Furthermore, the applicability of the methods depends only on the coincidence that an acid absorbing no iodine *usually* corresponds with an acid solid at ordinary temperatures, a noteworthy exception being the solid stereoisomeride of elæomargaric acid, the present writer having separated this constituent from china-wood oil solidified by exposure to light, finding its potash soap to be practically insoluble in 90 per cent. acetone, whilst its iodine absorption was 157.

The method as I now use it is as follows: About 10 grms. of the dry fatty acids are weighed into a 150 c.c. CO_2 flask, 90 c.c. of anhydrous acetone added, the solution warmed to 25° C., and 10 c.c. of $\frac{N}{1}$ potassium hydroxide added in a thin stream with stirring. The flask is then corked and immersed in ice-water for three to four hours. At the end of this period complete precipitation will have occurred.

The contents of the flask are then thrown on to a 5 or 6 cm. Buchner funnel and washed with anhydrous acetone at 0° C. until the residue is quite colourless. The bulk of the acetone is then removed by sucking air through the filter-paper and pressing down to a solid cake by means of a spatula. The cake is then placed in a tared 150 c.c. beaker, the adhering soaps removed from the filter-paper, and the CO_2 flask washed out with a little warm dilute potash, since part of the precipitated acids exists in the free state and adheres tenaciously to the flask. The rinsings of this flask are then run into the beaker containing the bulk of the soaps, water added, the flask warmed until solution is complete, and the acids liberated in the usual way, the boiling being continued until the lower aqueous layers become transparent.

The solid acids are then filtered off as in the determination of the Hehner acids, the cake solidified by immersion in cold water, and the drained paper with its adhering acids transferred to the tared beaker and dried at 100° C. until of constant weight.

The liquid acids may be separated from the acetone filtrate by adding an equal volume of water, 30–50 c.c. ether, and acidifying with hydrochloric acid. The ether layer is then washed with faintly acid water, the acid assisting in the separation of the emulsion. The ethereal layer is then run off, evaporated, etc.

A trial of a comparison of the lead-salt method with the above method gave the following figures:

	Lead-Ether Method.	Acetone Method.
Liquid acids	– (lost)	91·0 per cent.
Iodine value of ditto	190·9	195·6
Saturated acids	7·72 per cent.	8·11 per cent.
Iodine value of ditto	27·5	17·5

Iodine value of liquid acids prepared by Tortelli and Ruggieri's method, 195·4.

(In the above case the acids had been prepared by liberation of the acids from the soaps under petroleum ether, the acids, in consequence, contained a small amount of soluble acids; hence the saturated and liquid acids obtained total to 99·1 per cent. instead of 100 per cent.)

It will thus be seen that not only do the solid acids show a lower iodine absorption in the case of the acetone method than in the other, but that the liquid acids from the former treatment actually show a higher iodine absorption than those obtained by the Tortelli-Ruggieri method, which treatment is stated to yield the highest iodine value obtainable, although the separation is by no means quantitative. Apart from the rapidity of the method, it has the advantage that the acids are considerably less oxidisable in the state of the potassium soaps than in the case of the lead soaps, thus enhancing the accuracy of the determination.

The method has been in use for very many determinations of saturated acids in raw oils, amongst which the following may be cited:

Linseed oil—Calcutta seed	9·1 per cent., of iodine value	16·4
„ „ Baltic seed	6·6 „ „ „	20·0
„ „ North-Western seed	6·0	
Soya bean oil	13·8	12·1
Para rubber seed oil	16·3	10·7
China-wood oil (varies considerably)	54·1	
Crude Menhaden oil	23·5	20·2

Comparisons between the amount of saturated acids in a raw oil with that found in the same oil when oxidised till solid, have shown that unless precipitation of the latter as lead soap of saturated acid has occurred through the employment of a lead drier, no diminution of saturated acid occurs during the process of oxidation.

The preparation of the acids for the acetone-soap method in the case of oxidised oils consists in saponifying the oil with alcoholic potash, evaporating off the alcohol completely, taking up with water, and liberating the acids under petroleum ether with hydrochloric acid (Fahrion's method of estimating "oxidised acids"). Re-solution and re-precipitation of the oxidised acids should be resorted to if their percentage be high, as they are liable to entrap the unoxidised fatty acids. The petroleum ether solution is evaporated to dryness, and a convenient quantity weighed out for treatment with acetone, etc.

It may be mentioned that the above, and also the lead-salt-ether method, fails in the case of polymerised oils, since both the lead salt of the acids that have suffered polymerisation and the potassium soap of the same, behave towards their respective solvents in the same way as acids of the stearic series. These acids are at present undergoing examination, but it may be mentioned that they differ considerably in appearance from true saturated acids, and, moreover, have high iodine values.

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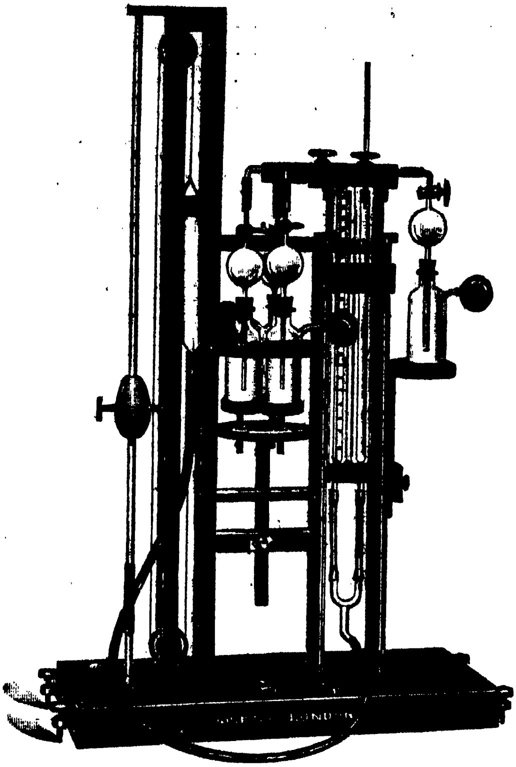
IMPROVED APPARATUS FOR THE ANALYSIS OF MIXED GASES.

By A. C. WILSON.

The accompanying illustration shows a modification of a well-known form of apparatus originally suggested by Dr. Stead for the analysis of blast furnace and producer gases, using mercury as a confining medium. In its present form the apparatus will be found to fulfil the requirements of the technical laboratory, and it is especially adapted for coal, water, and producer gases. The construction of the absorption pipettes is such as to provide against the deterioration of the contents through contact with the atmosphere or the fumes of the laboratory.

To those familiar with the usual patterns of "gas analysis apparatus" in general use, the illustration will convey a full impression of its aim and construction, and little further explanation is necessary, unless it be to point out that the bulbs shown as attached to the absorption bottles are in the form of thin gas bags, which readily expand when the air from the bottle is allowed to enter them. Combustion of the mixture is effected in the graduated eudiometer tube, which is provided with platinum terminals. If the palladium tube or other similar means be adopted, the platform on the right of the illustration is made use of, and the pipette shown replaced by the modified palladium tube.



This form of apparatus has been in regular use in the writer's laboratories for two years, and the modifications have been found to meet the whole of the difficulties experienced in using the older forms of the apparatus.

The substantial way in which it has been built render it much less subject to accident and more convenient to work.

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

A Rapid and Simple Method for the Estimation of Alcohol in Tinctures. G. Mossler and F. Markus. (*Zeits. Allgem. Oesterr. Apoth.-Ver.*, 1914, 52, 197-200, 215-217; through *Chem. Zentralbl.*, 1914, II., 89-90.)—The estimation of the alcoholic strength of tinctures by distillation does not afford satisfactory results; tinctures containing volatile oils show too high results, those containing resinous matters too low. The authors describe a method based on the critical solution temperature of a mixture of the alcoholic tincture and benzene. Ten c.c. of the tincture at 15° C. are measured into a tube together with 2 c.c. of benzene; the tube is closed with a cork through which passes a thermometer graduated in half-degrees. The tube is warmed in hot water until a perfectly clear mixture is obtained, and then allowed to cool, with frequent shaking, to the point at which a turbidity appears. The critical temperature is higher the weaker the alcohol; a difference of 1° C. corresponds to about 0·2 per cent. of alcohol. For liquids which would show a critical point above 60° C., the quantity of benzene is reduced to 1·5 c.c. The method is not suitable for tinctures prepared with 90 per cent. alcohol, nor for tincture of rhatany, on account of its colour. The influence of the non-volatile residue of the tincture on the critical solution temperature may be neglected. Every 1 per cent. of dry residue raises the sp. gr. of the alcohol contained in the tincture by about 0·0033. Tinctures of calamus and strophanthus show an exceptionally low coefficient; in all the other tinctures the coefficient ranges from 0·0030 to 0·0037. The authors' method may also be applied for the estimation of methyl alcohol in tinctures, since methyl alcohol shows a much higher critical solution temperature than ethyl alcohol. For instance, ethyl alcohol of 62·2 per cent. strength by weight shows a critical temperature of 26·5° C.; methyl alcohol of similar strength shows 82° C. It is thus possible to detect the presence of 4 per cent. of methyl alcohol.

J. F. B.

Analytical Characters of Benzoin. T. T. Cocking and J. D. Kettle. (*Brit. Pharm. Conference*, 1914, 13.)—To determine the dry alcoholic extract, which contains substances volatilising on evaporation, the moisture, found by drying the drug over sulphuric acid in a vacuum, is deducted from the dried residue obtained on extracting a fresh portion with 90 per cent. alcohol. The benzoic and cinnamic acids are estimated as follows: The alcoholic extract from 5 grms. of the material is saponified with potassium hydroxide, dried, dissolved in 100 c.c. of water, and the solution, acidulated with hydrochloric acid, is boiled with 5 grms. of light magnesium oxide and 20 c.c. of xylene under a reflux condenser for one hour; twice in succession the aqueous portion is cooled, filtered, and the filter and contents boiled with 100 c.c. more water. The filtrates are washed with ether, rendered acid with hydrochloric acid, and extracted with ether; the ethereal extract is concentrated on the water-bath, and the residual balsamic acids dried in a vacuum and weighed. No resin acids are extracted. The acids are allowed to stand overnight with a 5 per cent. solution of bromine in carbon tetrachloride, the excess of which is removed by evaporation on the water-bath with ether; the residue is dried as before and weighed. One molecule of cinnamic acid combines with 2 atoms of bromine. The free balsamic acids are determined as above, without the saponification. Analyses of 11 samples of benzoin are given. O. E. M.

Estimation of Strychnine in Presence of Brucine. D. B. Dott. (*Brit. Pharm. Conference*, 1914, 8.)—One c.c. of strong nitric acid added at 17° C. to 10 c.c. of the dilute sulphuric acid solution of the alkaloids containing 0·25 grm. brucine destroys all the brucine in twenty minutes (*cf.* Howard, ANALYST, 1905, **30**, 261). O. E. M.

Estimation of Caffeine in Coffee. G. Fendler and W. Stüber. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1914, **28**, 9-20.)—Examination of certain methods for the estimation of caffeine showed that those proposed by Lendrich and Nottbohm (ANALYST, 1909, **34**, 214) and by Katz (*ibid.*, 1902, **27**, 359) yield trustworthy results; Keller's method was found to be less accurate. Since the two first-mentioned methods are somewhat tedious in application, the authors give the following process and record results of estimations showing that it is accurate: Ten grms. of the powdered sample are shaken for thirty minutes with 10 grms. of 10 per cent. ammonia and 200 grms. of chloroform; the solution is then poured on a large filter, and 150 grms. of the filtrate are evaporated to dryness. The residue is digested for ten minutes with 80 c.c. of hot water, cooled, and 20 c.c. of a 1 per cent. potassium permanganate solution are added (10 c.c. are sufficient in the case of raw coffee). After fifteen minutes, a slight excess of hydrogen peroxide (3 per cent. hydrogen peroxide containing 1 per cent. of glacial acetic acid) is added, the mixture is heated for fifteen minutes on a water-bath, more hydrogen peroxide is added until the solution becomes clear, and then cooled and filtered, the filter and its contents being washed with cold water. The filtrate is shaken first with 50 c.c. of chloroform, then with three successive quantities of 25 c.c. of the solvent, the united chloroform extracts are evaporated to dryness, and the residue of caffeine is dried at 100° C. and weighed. W. P. S.

Determination of Cane Sugar in Beet Molasses by a Modification of the Double Polarisation (Clerget-Herzfeld) Method, using Citric Acid. V. Staněk. (*Zeitsch. Zuckerind. Böhm.*, 1914, **38**, 429-440.)—In applying the double polarisation method to beet molasses, the direct reading is taken in an alkaline solution (owing to the excess of basic lead acetate remaining after clarification), and the inversion reading in an acid medium; the difference between the two observations is not due solely to cane sugar, since the rotation of the optically-active impurities is modified by the change of reaction to an appreciable extent (ANALYST, 1913, **38**, 281-282). In order to avoid this error, the author proposes to make the direct polarisation in the presence of the same amount of citric acid as the inversion observation.

Preparation of Reagents.—To 380 grms. of pure potassium hydroxide are added about 250 c.c. of water, and, when three-quarters have dissolved, the solution (1) is poured off, the remaining hydroxide being dissolved in 250 c.c. of water (2). Four hundred grms. of citric acid are mixed with 100 to 200 c.c. of water, and solution 1 gradually added; the resulting solution is cooled, diluted to about 750 c.c., and neutralised by the carbonate-free solution 2 with phenolphthaleïn as indicator, being finally made up to 1,000 c.c. This is the neutral potassium citrate solution, 10 c.c. of which, on combination with the 5 c.c. of hydrochloric acid (sp. gr. 1·188) used for inversion, form potassium chloride and free citric acid. Five hundred c.c. of it are mixed with 250 c.c. of hydrochloric acid (sp. gr. 1·188), the liquid being cooled and made up to 1,000 c.c. This is the potassium chloride and citric acid solution, 20 c.c. of which contain the equivalent of the 5 c.c. of hydrochloric acid (sp. gr. 1·188) used for inversion.

Determination.—Twice the normal weight of the sample of molasses is dissolved in water, and made up to 200 c.c. without the addition of any clarifying agent. Direct reading: Fifty c.c. of this normal molasses solution are mixed with 20 c.c. of the potassium chloride and citric acid solution, made up to 100 c.c. with saturated bromine-water, mixed, filtered, and polarised in the 200 mm. tube. Inversion reading: Fifty c.c. of the normal molasses solution are inverted with 10 c.c. of a mixture of equal volumes of hydrochloric acid (sp. gr. 1·188) and water in the ordinary manner prescribed by Herzfeld, cooled, treated with 10 c.c. of the neutral potassium citrate solution, and made up to 100 c.c. with saturated bromine-water. After mixing, and allowing the liquid to stand for twenty minutes, it is polarised at a known temperature in a water-jacketed 200 mm. tube, the cane sugar content of the sample being calculated by the following formula:

$$\frac{(D-I)\,100}{142{\cdot}6-\frac{t}{2}},$$

D and I being the direct and inversion readings, and *t* the temperature in ° C. at which the inversion reading is observed. Determinations made with factory and refinery molasses showed that this method gives results that are in accord with the Andrlik-Staněk modification (ANALYST, 1913, **38**, 281), but may be about 1 per cent. higher than the ordinary procedure involving the alkaline direct reading.

J. P. O.

Colour Reactions for Citric Acid. E. P. Häussler. (*Chem. Zeit.*, 1914, **38**, 937.)—The liquid containing citric acid is evaporated to dryness with alcoholic vanillin solution; the residue is coloured violet when moistened with 2 or 3 drops of 25 per cent. sulphuric acid and warmed on the water-bath, and with water yields a green solution turning red with ammonia in excess. The reaction is sensitive to 1 to 2 mgrms. of citric acid. In the simultaneous presence of 0·02 grm. each of benzoic, salicylic, cinnamic, and teresantalic acid, or of 0·1 grm. each of succinic and lactic acid, with 0·2 grm. each of tartaric, malic, and lactic acid, the sensitiveness is reduced to between 20 and 50 mgrms. Sugar and proteins must be removed before the test is carried out. O. E. M.

Fat of Cohune Nuts. (*Bull. Imp. Inst.*, 1914, **12**, 237-242.)—The fat from the fruit and the kernels of cohune nuts from British Honduras gave the following analytical values:

	Fat from Fruit.		Fat from Kernels.				
	1	2	1	2	3	4	5
Sp. gr. 100/15·5° C. ...	0·848	0·855	0·870	0·871	0·871	0·868	0·870
Acid value	162·0	121·3	3·5	13·1	1·2	20·4	12·3
Saponification value ...	197·4	203·1	255·0	256·5	256·5	252·4	252·4
Iodine value	75·1	65·4	13·6	13·7	11·4	13·7	11·0
Hehner value	97·0	—	—	—	—	87·7	—
Reichert-Meissl value ...	1·65	—	6·8	8·3	8·2	7·1	—
Solid.-pt. of fatty acids ...	31·0° C.	—	19·8° C.	21·0° C.	20·2° C.	19·7° C.	—
Unsaponifiable matter, per cent.	0·95	—	0·24	0·28	0·23	0·28	—
Polenske value	—	—	—	15·4	—	12·5	—

The residual meal from the extracted kernels had the following composition, calculated on the basis of 10 per cent. of fat: Moisture, 9·2; crude proteins, 21·7; fat, 10·0; starch, etc., 38·8; fibre, 15·6; and ash, 4·7—per cent. Nutrient ratio, 1 : 2·9. C. A. M.

Copals from British West Africa. (*Bull. Imp. Inst.*, 1914, **12**, 217-222.)—The following values were obtained:

					Solubility.				
	Moisture.	Ash.	M.-Pt.	Acid Value.	Alcohol.	Ether.	Benzene.	Alcohol and Benzene.	Turpentine.
	Per Cent.	Per Cent.	° C.						
"Ozia" gum (*Daniella ogea*)	0·25	0·25	100–115	110–123	Insol. or partly sol.	Largely sol.	Insol. or slightly sol.	Nearly sol.	Insol. or partly sol.
Accra copal (*D. similis*)	0·8	0·08	115–180	102–129	Partly sol.	Partly sol.	Partly sol.	Sol.	Partly sol.

C. A. M.

Analysis of Essential Oils. A. Béhal. (*Bull. Soc. Chim.*, 1914, **15**, 565-568.)—Estimation of the esters in an essential oil may give results differing by as much as 3 per cent. This was attributed by Schimmel and Co. (*Bull.*, October, 1895) to the action of too concentrated a solution of alkali upon the alcohols. The author, however, shows that it is due to the fact that esters are decomposed in the presence of alkalis and alcohols, with the formation of a certain quantity of ester corresponding to the alcohol of lower molecular weight and the liberation of the alcohol of higher molecular weight. For example, the benzoic esters of various phenols and of cocaine when heated for a short time with alcoholic potassium hydroxide yield ethyl benzoate. In the ordinary process of saponification with an open tube as condenser, some loss of such volatile esters is inevitable, and to obviate this the saponification should be done beneath a reflux water condenser or in a sealed tube. C. A. M.

Essential Oils from Various Countries. (*Bull. Imp. Inst.*, 1914, **12**, 222-237.)

Oil.	Sp. Gr. at 15°/15° C.	Eugenol.	Optical Rotation $[\alpha]_D$.	Alde-hydes.	Sapon. Value.	Solubility in 70 per Cent. Alcohol.	Solubility in 80 per Cent. Alcohol.
		Per Cent.		Per Cent.			
Lemon grass, India	0·901–0·908	—	– 5° 35′	68·5–77	—	3 samples insoluble	Cloudy in 2 to 4 vols.
,, ,, "white-stemmed"	0·909	—	– 10° 50′ at 22° C.	9	—	Insol. in 5 vols.	Turbid in 4 vols.
,, ,, "red-stemmed"	0·925	—	—	71·5	—	Turbid with 5 vols.	Sol. in 0·8 vol., turbid in 5 vols.
Vetiver oil, Fiji	1·018	—	—	—	47	Turbid with 3 vols.	Turbid in 2·6 vols.
Oil of limes, Fiji	0·868	—	+ 37° 6′ at 21° C.				
Ylang-ylang, Seychelles ...	0·920	—	– 30° at 21° C.	Acid Value. 4·2	46·4	Sol. in 1 vol. 90 per cent. alcohol	
,, Mauritius ...	0·9883	—	– 30° at 20° C.	7	180	Sol. in 0·1 to 2·5 vols.	
Clove oil, Mauritius (2)... ...	1·061 and 1·067	89–95	– 0° 10′ and – 0° 5′ at 22° C.	—	—	Sol. in 1·5 vols.	
Clove-leaf oil, Mauritius (3) ...	1·059–1·062	90–91	– 0° 28′ at 22° C.	—	—	Sol. in 1·25 vols.	
Sandalwood oil (3)	0·9788–0·9814	—	– 20° 19′ to 21° 19′	1·4 to 2·2	2·8 to 4·7	Sol. in 5·5 vols.	

C. A. M.

Estimation of Lactic Acid in Wine by Moslinger's Barium Chloride Method. W. I. Baragiola and O. Schuppli. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1914, **27**, 841-881.)—The following modification of this method is recommended, since the process as described originally is liable to give inaccurate results, mainly owing to the reduction of barium chloride by carbon during incineration, to the retention of lactic acid by the alcohol-insoluble substances, and to the conversion of a portion of the lactic acid by malic acid into a compound in which the former acid is not estimated. Twenty-five c.c. of wine are mixed with 25 c.c. of water and steam-distilled until 200 c.c. of distillate have been collected; the distillate is treated with 5 c.c. of 10 per cent. barium chloride solution, and neutralised with saturated barium hydroxide solution. In case any lactic anhydride is present, an excess of barium hydroxide may be added, the solution heated for ten minutes on a water-bath, and then neutralised with hydrochloric acid, using azolitmin paper as indicator. The neutral solution is evaporated to 15 c.c., being kept neutral in reaction during the evaporation by the addition of barium hydroxide solution or hydrochloric acid, as required; the concentrated solution is transferred to a 100 c.c. cylinder, diluted with water to 25 c.c., and 95 per cent. alcohol is added drop by drop while the mixture is shaken continuously. The mixture is now diluted with alcohol to 100 c.c., placed aside overnight, and the volume again made up to 100 c.c. by the addition of alcohol (the first addition of alcohol causes the mixture to become heated, and contraction takes place on standing). The mixture is then filtered, 75 c.c. of the filtrate are treated with 25 c.c. of 5 per cent. sodium sulphate solution, the barium sulphate is removed by filtration, and 75 c.c. of the solution are evaporated to dryness; the residue is incinerated, the ash dissolved in water, and the boiling solution titrated with $\frac{N}{10}$ sulphuric acid solution, using azolitmin paper, methyl orange, or phenolphthaleïn as indicator. The number of c.c. of $\frac{N}{10}$ acid required is multiplied by 64 to give grms. of lactic acid per litre of wine. W. P. S.

Organic Phosphoric Acid Compounds in Concentrated Musts and Wines. C. Finzi. (*Staz. sperim. agrar. ital.*, 1914, **47**, 337-346; through *Chem. Zentralbl.*, 1914, II., 88.)—For the estimation of the organic phosphoric acid compounds in musts, 100 c.c. are evaporated to a syrup *in vacuo*, at 45° to 47° C.; the syrup is mixed with powdered glass or washed sand, and dried *in vacuo* over sulphuric acid at 45° to 47° C., with frequent stirring, until the mixture is capable of being pulverised. It is then extracted in a Soxhlet apparatus for twelve hours with dry ether, then with absolute alcohol, boiling under vacuum so that the temperature does not exceed 45° to 47° C. The alcoholic extract is evaporated *in vacuo*, the residue is oxidised by fusion with ten times its weight of a mixture of 3 mols. of potassium nitrate and 1 mol. of sodium carbonate; the melt is dissolved in dilute nitric acid, and the phosphoric acid estimated in the filtrate by double precipitation with molybdate solution and magnesia mixture. Lecithin $= Mg_2P_2O_7 \times 7{\cdot}2703$. In the case of wines, 500 c.c. are evaporated under reduced pressure at 48° C. with the passage of a current of dried air, the residue is dried *in vacuo*, mixed with sand, and extracted five times with 70 c.c. of ether and five times with 50 c.c. of absolute alcohol at 45° C.; the analysis is continued as described for musts. The author concludes that the organic phosphoric acid compounds of the musts are derived from the juice and flesh of the grapes, and are not produced by the action of the alcohol on the constituents of the marc. In the case of musts, no definite relation exists between the organic and inorganic phosphoric acid, but there appears to be a connection between the organic phosphoric acid and the nitrogenous compounds. In wines the proportion of organic phosphoric acid is much smaller. J. F. B.

Preservation of Milk Samples for Analysis. J. Tillmans, A. Splittgerber, and H. Riffart. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1914, **27**, 893-901.)—Mercuric chloride was found to be the most suitable substance for preserving milk samples for subsequent analysis. The addition of 0·04 per cent. of the salt keeps the milk quite fresh for 120 hours, has no influence on the methods used in milk analysis, and does not interfere with the detection and estimation of nitrates in the milk by means of the diphenylamine reaction. Thymol, phenol, mustard oil, sodium fluoride, chloroform, and potassium dichromate are unsuitable for the purpose. W. P. S.

Volumetric Estimation of Casein in Milk by Means of the Tetraserum. B. Pfyl and R. Turnau. (*Arbeit. Kaiserl. Gesundheitsamte*, 1914, **47**, 347-361.)—The method proposed depends on the acidimetric titration of the milk and of the tetraserum (*cf.* ANALYST, 1912, **38**, 450) obtained from it. Fifty c.c. of the milk are cooled to 15° C., 6 drops of 1 per cent. phenolphthaleïn solution are added, and the liquid is titrated with $\frac{N}{10}$ alkali solution. A further 50 c.c. of the milk are then treated with 5 c.c. of carbon tetrachloride, the mixture is shaken for ten minutes, 1 c.c. of 20 per cent. acetic acid is added (the strength of this acid must be accurately estimated), the mixture is again shaken and filtered. Twenty-five c.c. of the filtrate are then titrated, using phenolphthaleïn as the indicator. From the first titration and the strength of the acetic acid is calculated the amount of alkali required for 25 c.c. of milk *plus* 0·5 c.c. of acetic acid; let this quantity be a c.c. of $\frac{N}{10}$ alkali solution. Then, from the titration of the serum (b c.c. of $\frac{N}{10}$ alkali solution) and the fatty-content of the milk (grms. per 100 c.c.) the amount of casein is found from the equation—

$$\text{Casein (grms. per 100 c.c.)} = 0{\cdot}457\left(a - b.\frac{99{\cdot}3 - f}{100}\right).$$

The result may be expressed more accurately by taking into account the sp. gr. of the milk and of the tetraserum, but the difference is slight, amounting to about 0·02 per cent. only.

Pure casein separated from milk was found to contain 15·50 per cent. of nitrogen, and to have an equivalent of 1143. W. P. S.

Polarimetric Estimation of Lactose in Milk. E. Feder. (*Zeitsch. Untersuch. Nahr., Genussm.*, 1914, **28**, 20-29.)—Scheibe's method (ANALYST, 1901, **26**, 127) does not yield accurate results even when great care is taken in estimating the volume of the precipitated casein, etc.; moreover, the rotatory power of lactose is considerably influenced by the mercuric iodide and sulphuric acid employed to precipitate the proteins. When, however, the milk is curdled by the addition of asaprol, the precipitate consists essentially of proteins and fat, and its volume is approximately equal to the quantity of total solids less lactose and mineral matter. The following procedure is recommended for the estimation: 75 c.c. of the milk are treated in a 100 c.c. flask with 6 c.c. of a reagent prepared by dissolving 75 grms. of asaprol and 75 grms. of citric acid in hot water and diluting the solution to 250 c.c. The treated milk is then diluted to 100 c.c., shaken, allowed to stand for fifteen minutes, and filtered. The filtrate is now polarised at 20° C., in a 200 mm. tube, using sodium light; under these conditions each degree of rotation observed is equivalent to

0·9518 grm. of lactose. The reading observed is multiplied by 0·951 ; let this result be a, then the actual quantity of lactose, L, present is found from the equation—

$$L = a \frac{100{\cdot}8 - t + \frac{(96{\cdot}7 - f)a}{75}}{75}$$

where t is the percentage of total solids, and f the percentage of fat in the milk.
W. P. S.

Determination of the Viscosity of Milk as a Means of detecting the Addition of Water. W. D. Kooper. (*Milchw. Zentralbl.*, 1914, **43**, 169-179, 201-208; through *Internat. Inst. Agric. Monthly Bulletin*, 1914, **5**, 815-817.)—The viscosity is determined by using a burette-shaped apparatus, and the time of outflow expressed in seconds, of 10 c.c. of milk being divided by the time of outflow of 10 c.c. of water to obtain a value termed the viscosity constant, V; this value is about 1·588, and the percentage quantity of total solids, T, is found approximately from the equation—$T = \frac{V}{0{\cdot}1384}$. Under certain conditions, watered milk may give the same viscosity constant as pure milk, in which case the value obtained for T is too high. The greater the amount of added water in the milk the greater is the difference between the real and the calculated values for the total solids, and this difference affords a means of detecting the presence of added water. W. P. S.

Peru Balsam. K. Dieterich. (*Ber. deutsch. Pharm. Ges.*, 1914, **24**, 233; through *Chem. Zentralbl.*, 1914, I., 2207.)—From the steam distillation under 1·5 atmospheres of 6 kgrms. of pure Peru balsam there were obtained in three days 300 grms. of a yellow oil, smelling of Peru balsam, and having an acid value 13·80, saponification value 233·48, and iodine value 44·08. It gave neither the Dieterich zone reaction nor the blue Müller coloration. The residue from the distillation differed little from the original substance; its acid and iodine values were somewhere lower, its saponification value somewhat higher, and its cinnamein content unchanged. It gave a bluish-green instead of a golden-yellow nitric acid reaction, a less distinct Müller reaction, but the same Dieterich zone test. A sample of Peru balsam fifty years old gave normal values, but abnormal nitric acid and zone reactions, due, probably, not to adulteration but to the mode of preparation of the balsam. O. E. M.

Detection of Small Quantities of Rye Flour in Wheat Flour. R. Fanto. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1914, **28**, 79-83.)—When 10 grms. of rye flour are shaken for two and a half hours with 100 c.c. of water, then submitted to centrifugal action for one hour, and the viscosity of the liquid portion determined, the liquid will be found to have a much greater viscosity than that obtained from wheat flour under similar conditions. In the viscometer used by the author, a definite volume of water passed through the orifice in 51 seconds; with the liquid from wheat flours the time taken varied from 56 to 71 seconds, whilst the liquids from various rye flours required from 133 to 709 seconds. All the determinations of the viscosities were made at 17° C. The presence of from 2 to 3 per cent. of rye flour in wheat flour may be detected by this method. W. P. S.

Composition of Rums. Bonis. (*Ann. Falsif.*, 1914, **7**, 305-322.)—Results of analyses of 186 samples of rum are recorded, the samples representing rums from Martinique (124), Guadeloupe (45), Reunion (13), Marie-Galante (2), Cochin China (1), and Jamaica (1). The author considers that it is not possible to distinguish by the usual methods of analysis between rums made from molasses and those from sugar-cane juice. A minimum of 250 grms. of "impurities" per hectolitre of alcohol is suggested for pure rums. W. P. S.

Detection and Estimation of Saccharin. S. Condelli. (*Staz. sperim. agrar. ital.*, 1914, **47**, 308-316; through *Chem. Zentralbl.*, 1914, II., 85-86.)—The following reagents are required: (*a*) A saturated solution of magnesium sulphate and oxide prepared by boiling the constituents with water; (*b*) sodium tannate solution freshly prepared by dissolving 2 grms. of tannic acid in 10 c.c. of 2·5 per cent. sodium hydroxide solution; (*c*) magnesium oxide free from carbonate; (*d*) petroleum spirit, b.-pt. 100° to 120° C.; (*e*) freshly distilled ether; (*f*) a mixture of equal parts of ether and light petroleum, boiling below 70° C., freshly distilled; (*g*) sulphuric acid, about $\frac{N}{10}$ concentration; (*h*) a cold saturated solution of potassium permanganate; (*i*) 96 per cent. alcohol. Two methods are described for the estimation of saccharin:

Method 1.—In the case of solid preparations, 10 to 50 grms. are triturated with a sufficient quantity of solution (*a*) to give 100 to 150 c.c. of filtrate. The mixture is stirred on the water-bath and the reaction tested, more of solution (*a*) being added if necessary, and heating being continued for half an hour. In the case of liquid preparations, 10 to 100 c.c. are saturated with magnesium sulphate in the cold, then warmed on the water-bath, and treated with magnesium oxide to a distinct alkaline reaction. The liquid is clarified, if necessary, by the addition of a few c.c. of solution (*b*), the action of which may be assisted by a little alcohol. After diluting to a known volume, 50 to 100 c.c. of the filtrate are treated in a separating funnel with 20 to 30 c.c. of sulphuric acid, and shaken out three times with an equal volume of the ether and light petroleum mixture. The evaporated extract is dissolved in dilute sulphuric acid, treated on the water-bath with permanganate drop by drop until the pink colour is permanent, and the filtrate is shaken out three times with freshly distilled ether. The evaporation-residue of the ethereal extract is saccharin.

Method 2.—In the presence of large proportions of fat, ethereal oils, alcohol, or other substances which dissolve saccharin, 25 to 50 grms. of the sample are placed in a separating funnel, shaken thoroughly with 100 c.c. of solution (*a*), heated to 50° to 60° C., and shaken vigorously with 100 c.c. of the petroleum spirit (*d*). After standing for some hours, the aqueous portion is filtered into a second separating funnel and treated further according to Method 1. J. F. B.

Fluorine in Mineral Waters. A. Gautier and P. Clausmann. (*Comptes rend.*, 1914, **158**, 1631-1640.)—The fluorine in various mineral waters was estimated by precipitation together with barium sulphate, or in some cases with magnesium ammonium phosphate, according to the method previously described by the authors (ANALYST, 1912, **37**, 517). Fluorine occurs in all mineral springs, hot or cold, in quantities ranging from 0·15 mgrm. (Eau d'Evian) up to 6 mgrms. (Eau de Vichy, Hôpital) per litre. The springs richest in fluorine rise from volcanic sources, those waters characterised by sodium bicarbonate being particularly rich. Of cold springs of surface origin, those containing calcium sulphate may contain over 2 mgrms. of fluorine per litre, since calcium fluoride is generally co-deposited in gypseous strata. In waters of similar origin, the proportion of fluorine bears no relation to the temperature of the spring. In waters of the same origin, whether thermal or not, the proportion of fluorine, although not proportional to the total salinity, generally varies with it. The sulphurous waters possess the highest fluorine-content in relation to their saline residue. Waters of the same hydro-mineral group, even from the same locality, may show large differences in their fluorine-content. In sea-water the proportion of fluorine varies round about 0·3 mgrm. per litre. Seismic disturbances may profoundly affect the proportion of fluorine in the water of thermal springs, presumably by opening up or obstructing the sources or passages of the springs. J. F. B.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Delicacy of the Peroxydase Reaction. A. Bach. (*Ber.*, 1914, **47**, 2122-2124.)—An infusion of horse-radish was treated with an equal volume of alcohol, and the peroxydase was precipitated from the clear filtrate by the addition of 3 volumes of alcohol. The aqueous solution of this precipitate was subjected to ultrafiltration through a layer of collodion deposited in the meshes of a linen cloth supported on a perforated funnel in connection with a filter-pump. The colloidal deposit on the filter was washed as far as possible and then dissolved in water. This solution, containing 0·127 per cent. of dry substance, was employed for the study of peroxydase reactions. Per mgrm. of dry substance, it was far more powerful than any previous preparations. For determining the limits of delicacy of the enzyme reaction, guaiacol is a far better substratum than pyrogallol or quinol, because, in the absence of peroxydase, hydrogen peroxide produces no change even after long standing. In the experiments, 1 c.c. of peroxydase solution was mixed with 8 c.c. of a 0·1 per cent. solution of guaiacol and 1 c.c. of 0·1 per cent. hydrogen peroxide. With 1 part of peroxydase dry-substance in 500 million of water, a coloration appeared in five minutes; with 1 per 1,000 million a distinct, gradually increasing coloration was perceptible in twenty minutes, while 1 part in 2 billion produced a visible reaction in twenty-four hours. J. F. B.

Analysis of the Saline Matter adhering to the Flesh Side of Wet Salted East India Goat Skins. M. C. Lamb. (*Collegium*, 1914, 357.)—A portion of the salt earth cure removed from the skins was subjected to gentle ignition to remove traces of organic matter, and was then found to have the following percentage composition: Silica and sand, 8·1; SO_4, 48·6; Cl, 9·9; Mg, 2·5; Ca, 2·2; Fe, 1·8; Na, 23·8; K, 3·0. The cure thus consists chiefly of sodium sulphate, though a little sodium chloride is also present. Proctor and Towse (*J. Soc. Chem. Ind.*, 1895, **14**, 1025) found no sodium chloride present on the flesh side of Ducca kips imported in the dry state. The hygroscopic nature of the "cure," which is added at the rate of 4 to 6 ounces per skin, adds considerably to the weight of the skins which are sold by weight. The moisture in the skins was about 20 per cent. H. F. E. H.

ORGANIC ANALYSIS.

Microchemical Reactions of Certain Anthraquinones. E. Senft. (*Zeitsch. Allg. Oesterr. Apoth.-Ver.*, 1914, **52**, 165-166; through *Chem. Zentralbl.*, 1914, II., 82-83.)—*Rhodocladonic acid* dissolves in sodium carbonate or bicarbonate with a red-brown coloration, this colour is discharged by potassium permanganate in the cold with formation of a reddish precipitate. The acid is insoluble in most ordinary solvents. Concentrated sulphuric acid dissolves it with intense purple-red coloration; the latter quickly fades to yellow, and the rhodocladonic acid separates in the form of very characteristic thin plates, rounded at both ends, often of oval, elliptical or circular shape, which, when set up on edge, have the appearance of thin needles. Besides these, dagger-shaped pointed needles are observed. The foxy-red colour of the crystals is also characteristic. The author has identified this acid in a number of *Cladonia* species.

Solorinic acid sublimes with yellow fumes at a temperature considerably higher than its melting-point. The sublimate is dichroic, appearing greenish-yellow against a black ground and reddish against a white one. Under the microscope it appears as amorphous, nearly colourless particles, which soon change into thin needles of uniform width obliquely cut off at both ends, of a pale yellow colour. It is soluble in sulphuric acid with a violet coloration, soon changing to yellow, with the separation of a flocculent amorphous precipitate, which gradually takes the form of short orange or yellow rods; these often adhere together in the form of chains. The direct identification of solorinic acid in lichens is difficult, but is easily performed on the sublimate from the lichen. In 10 per cent. potassium hydroxide the small crystals are coloured red, and finally violet; they do not dissolve, but on heating break down into an amorphous violet precipitate.

Rhodophyscin, contained in the lichen *Physcia endococcinea*, crystallises from boiling acetic acid very indistinctly in bunches of small crystals; it does not sublime. Its solution in sulphuric acid is first purple, then dirty brown-red, and finally green, with separation of a brownish-green precipitate. With 10 per cent. potassium hydroxide the solution is raspberry-red, forming later an amorphous violet precipitate.

Blastenin is readily soluble in chloroform; it yields a yellow sublimate, non-dichroic, consisting of yellow droplets and minute, nearly colourless, crystals. The latter are only partly soluble in potassium hydroxide, and the droplets are coloured carmine, but do not dissolve. The sulphuric acid solution is carmine-red, slowly changing to yellow, with separation of colourless or yellow needles often in bundles. Blastenin is readily soluble in hot bone oil or paraffin, from which it crystallises slowly in bundles of yellow or orange needles. J. F. B.

Analysis of the Asphalts. J. Marcusson. (*Chem. Zeit.*, 1914, **38**, 813-815, 822-823.)—The difference between natural asphalts and mineral oil pitches is determined by pouring the benzene solution of the sample into petroleum spirit, filtering off the precipitate, shaking the filtrate with sulphuric acid, and testing the oily residue obtained. Natural asphalts yield about 17 per cent. of oily matter, fluid at the ordinary temperature, and poor in solid paraffins. Petroleum asphalts yield larger quantities (20 to 50 per cent.) of oily matter, semi-solid at 20° C., and containing over 2 per cent. of solid paraffin. Against criticisms by Loebell (ANALYST, 1914, 131), the author points out that, according to his method, the solid paraffin is estimated by precipitation with ether alcohol from an oil which is distilled after separation from the pitchy matters by solvents and sulphuric acid. Loebell's numbers relate to the paraffin-contents of oils distilled in presence of the pitch, in which case overheating and "cracking" of the solid paraffins reduce the amount determined. It is, however, conceivable that in exceptional cases petroleum asphalts might be produced, yielding an oil with less than 2 per cent. of paraffin. In such cases the quantity of the oil serves as a guide. Further, the saponification value of natural asphalts ranges round about 30; that of petroleum asphalts about 10. The percentage of sulphur may be a useful indication, if the Mexican and Californian petroleum residues, rich in sulphur, be excluded. Roumanian products contain less than 1 per cent. Lastly, the distillates from natural asphalts show a distinct acidity, whilst those from petroleum products are nearly neutral.

For estimating the acid value of the asphalts, 10 grms. of the bitumen are dissolved in 25 c.c. of benzene; the solution, after cooling, is poured into 100 c.c. of neutral alcohol, and allowed to stand overnight. The clear liquid is decanted off, and the residue washed with 50 c.c. of alcohol; the total extract is then titrated with $\frac{N}{10}$ sodium hydroxide in presence of alkali-blue. The acid values range from 2·8 to 15·4. Trinidad and Bermuda asphalts give the highest numbers, while the low values are obtained with bitumens derived from asphalt-sands, in which the acids are partly combined with calcium. Petroleum asphalts show acid values of 0·1 to 0·4. Asphalts refined by prolonged fusion show lower values than the same products in the crude state; the acidity falls on heating, mainly owing to anhydride formation. The bodies which undergo saponification with boiling alcoholic soda appear to consist of anhydrides of the acids, not esters or waxes; the acids liberated are resinous even after distillation of the asphalt, and are almost insoluble in petroleum spirit. On the other hand, the resinous bodies in oxidised fatty oil pitches are converted into ordinary fatty acids when the pitch is distilled. The acids of natural asphalt show a close similarity to the polynaphthenic acids formed by the resinification of mineral oils on prolonged heating at 120° C.

Asphaltites differ from asphalts and pitches in being brittle and easily pulverised. The acid value is generally less than 1, with the exception of manjak (2·4). The sulphur is much lower than in natural asphalts; the mineral matters also are very small. The oily residue is generally below 7 per cent., manjak (17 per cent.) being an exception. The saponification value is low—about the same as for petroleum asphalts.

For the determination of the melting-point, the author recommends the Krämer-

Sarnow method (*cf.* Schanin, ANALYST, 1914, 264)—penetration of a drop of mercury through a layer of asphalt of prescribed thickness. The values coincide fairly closely with the point "beginning to flow" as determined by the Ubbelohde method; and this latter corresponds, in the case of ceresins, with the beginning of melting in a capillary tube. Ubbelohde's point "beginning to drip" is situated some 15° to 20° C. higher, and corresponds to the end of the melting in a capillary tube tested on ceresins. J. F. B.

Estimation of Benzoyl in Benzoyl Derivatives. A. W. van der Haar. (*Arch. Pharm.*, 1914, **252**, 205-208.)—About 0·5 grm. of the dried benzoyl derivative is saponified with alcoholic alkali beneath a reflux condenser, the alcohol evaporated on the water-bath, and the residue acidified with phosphoric acid, and repeatedly shaken with ether. The united ethereal extracts are dried with anhydrous sodium sulphate, and concentrated to a small residue at a low temperature on the water-bath. The remaining ether is allowed to evaporate spontaneously, the residue dried at 40° C. to constant weight, and then heated in an oven at 115° to 120° C., until the weight again becomes constant. The loss in weight gives the amount of benzoic acid in the benzoyl derivative. Analyses of various benzoyl compounds are cited to show that the method gives accurate results, provided that the compound is soluble in ether, and that only the benzoic radicle is sublimed from it at 115° C.

C. A. M.

Estimation of Berberine. E. Richter. (*Arch. Pharm.*, 1914, **252**, 192-205.)—A gravimetric method of estimation is based upon the facts that berberine is quantitatively precipitated by sodium hydroxide, and that, on extracting it with ether and adding picrolonic acid to the ethereal extract, berberine picrolonate—

$$C_{20}H_{18}NO_4.C_{10}H_8(NO_2)_2N_2O—$$

is quantitatively precipitated. An aqueous solution of berberine chloride (about 0·1 grm. in 20 c.c.) is treated with 10 c.c. of 15 per cent. sodium hydroxide solution, and the liquid shaken with 60 grms. of ether. Forty grms. of the ethereal extract are shaken with 5 c.c. of approximately $\frac{N}{10}$ picrolonic acid, and the precipitate collected after an hour in a Gooch crucible, washed with alcohol-ether (3 : 1), dried at 110° C., and weighed. The weight, multiplied by the factor 0·561, gives the amount of berberine. In estimating berberine in the tincture of *Berberis vulgaris*, 40 grms. of the sample are diluted with a little water, treated with 10 c.c. of 15 per cent. sodium hydroxide solution, and shaken for ten to fifteen minutes with 80 c.c. of ether. About 1 grm. of powdered gum tragacanth is then added, the shaking continued until the ethereal layer is clear, and the berberine precipitated as described. For a volumetric estimation, a definite quantity of the ethereal solution is shaken with 10 c.c. of $\frac{N}{10}$ picrolonic acid solution (previously standardised on berberine), made up to 100 c.c., allowed to stand for thirty minutes, and filtered. Fifty c.c. of the filtrate are mixed with 50 c.c. of alcohol, and the excess of picrolonic acid titrated with $\frac{N}{10}$ alcoholic alkali with Poirrier's blue as indicator. Each c.c. of $\frac{N}{10}$ picrolonic acid in water or alcohol corresponds to 0·0336 grm. of berberine. The results are less accurate than those given by the gravimetric method. C. A. M.

Action of Solvents on Coal. L. Vignon. (*Bull. Soc. Chim.*, 1914, **15**, 540-544.)—The following amounts of substances were dissolved on treating 10 grms. of Montrambert coal for three hours with the different solvents at the ordinary temperature: Alcohol (96 per cent.), 0·076; ether, 0·059; benzene, 0·080; toluene, 0·078; aniline, 2·250; and nitro-benzene, 1·41 per cent. At the boiling-points of the solvents, aniline dissolved 12·05 and nitro-benzene 3·19 per cent. Three samples of Loire coal, representing types rich and poor in gas, and medium in this respect, gave the following results on extraction with boiling aniline:

	Soluble Constituents, less Ash.	Soluble Constituents.
	Per Cent.	Per Cent.
Coal rich in gas	23·40	26·8
Coal, medium	6·58	7·2
Coal poor in gas	1·56	1·8

The constituents soluble in aniline can be precipitated by acids. They contain more hydrogen and less ash than the insoluble substances, and yield a solid spongy coke instead of a powdery coke. Coals rich in gas are also soluble to a considerable extent in boiling quinoline (*cf.* ANALYST, 1914, 320). C. A. M.

Comparison of Various Methods for determining the Iodine Value of Fats. St. Weiser and H. C. Donath. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1914, **28**, 65-73.)—As the result of an investigation, the author prefers Winkler's bromate method for determining the iodine value of fats, oils, and fatty acids. In this method a weighed quantity of the fat is dissolved in 10 c.c. of carbon tetrachloride, 50 c.c. of $\frac{N}{10}$ potassium bromate solution and 1·5 grms. of potassium bromide are added, the mixture is acidified with 10 c.c. of 10 per cent. hydrochloric acid, and then set aside in a dark place for two hours (in the case of fish oils, contact for four hours is required). The flask containing the mixture must be tightly stoppered. Fifteen c.c. of 10 per cent. potassium iodide are then added, and the liberated iodine is titrated with thiosulphate solution. The results obtained agree closely with those found by the Hübl, Waller, and Wijs methods. (See also ANALYST, 1911, **36**, 19.) W. P. S.

Physical Constants of Isoprene. C. Harries. (*Ber.*, 1914, **47**, 1999-2003.)—Concordant results for the boiling-points of highly volatile liquids are only obtained by heating in a bath a few degrees above the boiling-point and distilling at a slow speed. In fractionating samples of isoprene, the author used a water-bath at 36° to 37° C., distilling at the rate of 30 to 40 drops per minute. The boiling-point of pure isoprene is 33·75° to 34° C. under a pressure of 762 mm. The density at $\frac{16{\cdot}5^\circ}{4^\circ}$ is 0·6807, and the refractive index $[N]_{D\,16{\cdot}5^\circ\,C.}$ is 1·42617. The above constants were obtained with a synthetic isoprene prepared from the hexamethyl-ammonium base by the Elberfeld Company. Another sample, prepared by the Badische Company, left 4 per cent. of an oily residue on preliminary distillation over sodium; the density of the main fraction, corresponding to pure isoprene in boiling-point and elementary composition, was very slightly lower, and the $[N]_{D\,15{\cdot}5^\circ\,C.}$ 1·42317. The latter constitutes a difference outside the limits of experimental error, and it is suggested that this second preparation either contains an impurity or represents a physical isomeride of ordinary isoprene. J. F. B.

Determination of Malt Extract. W. P. Dreaper. (*J. Soc. Dyers and Col.*, 1914, **30**, 255-257; through *J. Soc. Chem. Ind.*, 1914, **33**, 786.)—The farina solution is padded into the cloth by passing through rollers, and the solution is of such a strength that the cloth contains only 2·5 per cent. of starch (dry). Errors due to the presence of moisture are avoided by estimating the moisture in the cloth at the time of the assay by drying at 100° to 102° C. The treated samples are also dried at this temperature. The treated samples are preferably wrung by hand, and not in a cloth. Drying is effected first in a porcelain dish, then in a weighing tube. Blank experiments under these conditions showed no loss of starch. The present procedure is as follows: Pieces of starched cloth (2 to 3 grms.) are immersed in the malt extract solution of standard strength. The ratio of cloth to solution should be 1:200. The time of immersion may vary from five to ten minutes, provided it is the same in all cases. The temperature may be the standard one of 40° C. or that used in practice. If the amount of starch removed varies greatly from 1·5 to 1·8 per cent., it will be better to vary the strength of the solution in a second assay. A correction of 0·2 per cent. may be made on account of loss of starch due to water alone. The total starch on the cloth is determined by treating it with malt extract at 40° C. for one hour, with constant stirring, drying at 100° to 102° C., and weighing. Objections to this method on the grounds of variations in conditions, etc., due to the "personal equation," were shown to be invalid, as what is required in the textile industry is simply a comparison of value for starch-removing purposes. Such minor differences were insignificant in view of the extraordinary variations observed between the different extracts at present on the market. It was stated that two samples of extract might easily vary as much as 50 per cent. in their rate of action on gelatinised starch in solution or on a fabric.

Detection of Methyl Alcohol. A. Rinck. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1914, **28**, 98-99.)—The alcoholic solution to be tested is placed in a flask closed with a cork, through which passes a silica tube. The portion of the latter above the cork is bent downwards at an angle of about 60°, and a copper spiral is placed in the inclined part of the tube. The spiral is heated by a burner placed under the tube, and the liquid in the flask is distilled slowly, the distillate being collected in a cooled receiver. The first few drops of distillate are tested for formaldehyde by the sulphuric acid morphine test. W. P. S.

Methods for the Estimation of Mannitol. J. Smit. (*Zeitsch. anal. Chim.*, 1914, **53**, 473-490.)—Hitherto two methods have been used for this purpose, that of Gayon and Dubourg (*Ann. de l'Inst. Pasteur*, 1894, **8**, 108; 1901, **15**, 527) and that of Müller (*Bull. Soc. Chim.*, 1894 [3], **11**, 329, 1073.)—The first method consists essentially in concentration of the solution until mannitol crystallises, treatment of the magma with a saturated alcoholic solution of mannitol, followed by filtration, and final extraction of the mannitol from the dry contents of the filter by means of hot alcohol. The limitations of such a method are obvious, and are demonstrated in the present paper.

Müller's method depends on the fact that when an aqueous solution of mannitol (and of many other substances which are either optically inactive or very feebly active) is saturated with borax, the rotatory power is notably raised. Since all sugars, as well as tartrates and malates, are affected in a similar manner by borax, and since glycerol, if present, combines with some of the borax and minimises the effect of a fixed quantity of the latter, the estimation of mannitol in wine by this method is troublesome, involving fermentation of sugar, removal of tartrates and malates, and three separate experiments to correct for the effect of glycerol. Detailed directions are given in the present paper, and Müller showed that the results, in the case of wine, were very satisfactory. In presence of some other substances, such as yeast-water, often employed as nitrogenous food for yeast or bacteria in fermentation experiments, this method fails utterly, and the following method, which is of general application, has been worked out.

It is modelled on Wagenaar's method for the estimation of glycerol (*Pharm. Weekblad*, 1911, 497), and depends on the capacity possessed by polyhydroxy-alcohols of holding cupric hydroxide in solution. Ammonia, amino-acids, sugars, hydroxy-acids, and glycerol and other polyhydroxy-alcohols must be first eliminated. Only amino-acids with free amino-groups interfere. They are removed, together with ammonia, by means of phosphotungstic acid. Sugars are fermented away or destroyed by heating with caustic alkali. If much sugar is present, the solution may become highly coloured, and must then be decolorised with lead acetate and the excess of lead removed as sulphate. Lactic acid is removed by extraction with ether and tartaric acid as calcium or lead tartrate. Glycerol is separated by the ether-alcohol method. When large quantities of yeast-water are present, the influence of the constituents other than amino-acids may be considerable, and, for the most exact work, sugar should be added and the mixture fermented, when the greater part of the constituents of the yeast-water are eliminated. The only remaining substances which can interfere are other hexitols, such as sorbitol and dulcitol. In such cases the method breaks down, and resort must be had to Meunier's method (*Ann. Chim. Phys.*, 1891, **22**, 412), which depends on the formation of well-characterised acetals, when mannitol or sorbitol are treated with excess of certain aldehydes (paraldehyde, benzaldehyde) in presence of concentrated hydrochloric acid. Dulcitol does not react under Meunier's conditions. The method is far from quantitative, but is the only one available when a second hexitol is present in quantity.

In absence of other hexitols, 50 c.c. of the solution, freed from interfering substances, are mixed in a cylinder with 25 c.c. 4 N sodium hydroxide and 25 c.c. copper sulphate solution (125 grm. crystals per litre), shaken for a few seconds only (otherwise the precipitate will not settle), allowed to stand, and 25 c.c. of the clear supernatant liquid are pipetted off, mixed with excess of potassium iodide and 10 c.c. of 25 per cent. sulphuric acid, and titrated with $\frac{N}{10}$ thiosulphate. The latter is the exact measure of the unprecipitated copper, but not of the amount of mannitol originally present, which must be derived from a curve or table based on direct experiment. Both curve and table are given in the paper. The following is an abbreviation of the table. The right-hand columns give the number of mgrms. of mannitol in the 100 c.c. of mixture in the cylinder when 25 c.c. of the supernatant liquid require the amounts of $\frac{N}{10}$ thiosulphate stated in the left-hand columns.

Thiosulphate.	Mannitol.	Thiosulphate.	Mannitol.	Thiosulphate.	Mannitol.
C.c.	Mgrms.	C.c.	Mgrms.	C.c.	Mgrms.
0·25	0·0	8	277	20	660
0·5	8·3	9	311	21	692
1·0	27·4	10	344	22	724
1·5	47·8	11	378	23	757
2·0	68·0	12	411	24	790
2·5	88·0	13	442	25	823
3·0	107·1	14	472	26	856
4·0	142·8	15	503	27	889
5·0	176·7	16	534	28	931
6·0	210·0	17	565	29	992
7·0	243·3	18	597	29·1	1000
—	—	19	628	—	—

G. C. J.

Microchemical Detection of Myrosin. K. Peche. (*Ber. d. Botan. Ges.*, 1914, **31**, 458-462; through *Chem. Zentralbl.*, 1914, II., 270.)—Potassium myronate is hydrolysed by myrosin with the formation of potassium bisulphate, and the latter may be identified by its reactions with the chlorides of alkaline earth metals. The section of the plant is immersed in a 10 per cent. solution of potassium myronate which has been saturated with barium, strontium, or calcium chloride. In the presence of myrosin the contents of some, but not all, of the cells will be coated with barium, strontium, or calcium sulphate. C. A. M.

Determination of Nitrogen in Guncotton by Means of the Nitrometer. E. G. Beckett. (*J. Soc. Chem. Ind.*, 1914, **33**, 628.)—The original Lunge method is compared with the modification in which 1 c.c. of water is added to the mixture of 15 c.c. of sulphuric acid and guncotton before the mixture is shaken. The gas evolved, the residual sulphuric acid and the sulphuric acid used were examined. By the first method, there were obtained from 5·1 grms. of guncotton 111 to 115 c.c. of gas, consisting of nitric oxide with 0·4 c.c. of carbon dioxide and sulphur dioxide together, 0·3 c.c. of carbon monoxide, and 0·6 c.c. of nitrogen. The residual sulphuric acid contained nitrogen equivalent to 0·75 c.c. nitric oxide, and the 15 c.c. of 96·5 per cent. sulphuric acid used gave up 0·35 c.c. of nitrogen during the estimation. The errors balance, so that the volume of gas measured represents correctly the nitrogen-content of the guncotton. The second method gave somewhat higher results, the gas evolved containing more carbon monoxide, carbon dioxide, and sulphur dioxide, due to heating on the addition of the water, and not to the use of more dilute acid. O. E. M.

Estimation of Ricinoleic Acid in Oils. F. Erban. (*Seifenfabrik.*, 1914, **34**, 493-495, 525-527, 556-557, 585-587; through *Chem. Zentralbl.*, 1914, II., 358.)—The acid value does not always give the amount of ricinoleic acid in an oil, owing to the tendency of this acid to condense with the formation of di- and poly-ricinoleic acids. Whilst the acid value of ricinoleic acid is 188, that of di-ricinoleic acid is 96·8. If, however, the acid anhydrides be saponified, the poly-acids are converted into the normal acid, and the correct acid value is ascertained by taking into account the altered weight of the fatty acid. Formulæ are given in the original paper for calculating the composition of mixtures consisting of ricinoleic acid and di-ricinoleic acid, of ricinoleic acid, di-ricinoleic acid, and neutral fat, of di-ricinoleic acid, lactide, and di-glyceride, etc. W. P. S.

Colour Reactions of Organic Compounds with Trichloroacetic Acid. K. Charitschkow. (*J. Russ. Phys.-Chem. Ges.*, 1914, **46**, 76; through *Chem. Zentralbl.*, 1914, I., 2202.)—Like tetranitromethane, which serves for the detection of double bonds, at least in cyclic compounds, trichloroacetic acid gives a colour-reaction with cholesterin. It was found to give a coloration with naphtha, petroleum, solar oil, turpentine, menthene, terebenthine, phenanthrene, mesitylene, colophony, colophony oil, and oleïc acid, but not with benzene hydrocarbons and some unsaturated compounds of low boiling-point. O. E. M.

Detection of Minute Quantities of Phlorrhizin Alone and in Presence of Phlorin. E. Griese. (*Beitr. Phys.*, 1914, **1**, 23-52; through *Chem. Zentralbl.*, 1914, II., 88-89.)—For the detection of minute quantities of phlorrhizin in solutions the author employed the reactions with ferric chloride (officinal solution diluted 1 : 100), with Millon's reagent (brownish-red precipitate or coloration), and with mercuric nitrate (white precipitate or turbidity). The limits of sensibility for the tests were reached with 1 c.c. of the following solutions: For the ferric chloride test, 0·00178 per cent.; for Millon's reagent, 0·00197 per cent.; for mercuric nitrate, 0·00042 per cent. The limit at which phlorrhizin can be extracted from aqueous solution by ether and amyl alcohol is 0·1 mgrm. per 100 c.c. By means of this extraction method it was possible to prepare phlorin free from phlorrhizin.

J. F. B.

Estimation of Rhamnose in Presence of Other Methylpentoses. E. Votoček and R. Potmešil. (*Bull. Soc. Chim.*, 1914, **15**, 634-639.)—The sugars most commonly found in glucosides, apart from dextrose, are—*d*-galactose, *d*-mannose, and members of the methylpentose group, generally rhamnose, often also rhodeose. Simple pentoses are rarely met with. Dextrose may be characterised by fermentation; galactose is precipitated almost quantitatively by methylphenylhydrazine, mannose by phenylhydrazine, arabinose by diphenylhydrazine. The method now described for the estimation of rhamnose depends on the formation of a cyanhydrin with hydrocyanic acid, which is easily converted by saponification into α-rhamnohexonic acid (or its lactone), which in its turn yields mucic acid on oxidation by nitric acid. For the estimation, 1 mol. proportion of rhamnose is treated with 2 mols. of hydrocyanic acid of moderate strength at the ordinary temperature, with the addition of a few drops of dilute ammonia to accelerate the reaction. After twenty-four hours the excess of hydrocyanic acid is removed by boiling, and the cyanhydrin decomposed by heating with about 1 mol. of sodium hydroxide. When the boiling liquid ceases to evolve ammonia it is evaporated to dryness on the water-bath. The residue is treated with 60 c.c. of nitric acid (sp. gr. 1·15), and the solution heated on the water-bath in a beaker of 60 mm. diameter at the bottom. After the liquid has evaporated to one-third of its original volume it is allowed to cool, and the sides of the glass are rubbed with a rod. After forty-eight hours the precipitate of mucic acid is collected in a Gooch crucible, washed with 20 c.c. of cold water, and dried in the oven. On the average, 45·5 parts of mucic acid correspond to 100 of anhydrous rhamnose, and the results show only slight variations. In presence of rhodeose, in equal quantity, the results ranged from 100·7 to 112·9 per cent. of the theoretical quantity of rhamnose, but since it is only necessary to determine the molecular proportions of a mixture of methylpentoses, the accuracy of the method is sufficient. J. F. B.

Identification of Rosin and Glue Sizings. W. Herzberg. (*Chem. News*, 1914, **110**, 19.)—Rosin is identified by the Raspail reaction, the production of a rose or violet coloration with sugar and concentrated sulphuric acid. Extraction methods with alcohol or ether are worthless. Animal size is identified by the precipitation of its solution with tannin, by its yielding a yellow or brown colour with iodine in potassium iodide solution, by the xanthoprotein reaction, the Millon test, or the biuret reaction; since it gives completely negative results with rosin sizes, the biuret reaction is to be preferred. These tests are best carried out under the microscope. Casein gives the Adamkiewicz test, a red-violet coloration with glacial acetic acid and sulphuric acid. O. E. M.

Examination of Bleached Shellac. H. Wolff. (*Chem. Rev. Fett.- u. Harz.-Ind.*, 1914, **21**, 159-161.)—Successive extraction of bleached shellac with petroleum spirit, ether and benzene yields a remarkably constant extract. This is reduced to a value of less than 1 by the presence of even a small percentage of colophony. The acid value of numerous samples ranged from 40 to 65 (70 and 76 in two exceptional cases); the saponification value, from 200 to 250 (268 and 275 in the exceptional samples); and the iodine value (Wijs) between 4 and 10. The presence of chlorine in organic combination is a characteristic of bleached shellac, and may be used as a test of its presence in other resins. The proportion of chlorine in 64 samples ranged from 0·8 to 2·4 per cent. The greater the quantity of chlorine the more likely is free hydrochloric acid to be liberated from the shellac, and to attack a lacquered surface. Free mineral acid is estimated by dissolving 1 to 2 grms. of shellac in 30 to 50 c.c. of alcohol and mixing the solution with 300 to 400 c.c. of water. A few drops of amido-azobenzene are added and the liquid titrated with $\frac{N}{10}$ sodium hydroxide solution until yellow, and the titration then completed after the addition of phenolphthaleïn. Shellacs suitable for lacquering should not show a "water-soluble acid value" of more than about 30, or about half to two-thirds of the acid value of the resin.

Certain commercial samples show a further development of free mineral acid in the aqueous solution after twenty-four hours, and would be unsuitable for some lacquers (*cf.* ANALYST, 1914, 139). C. A. M.

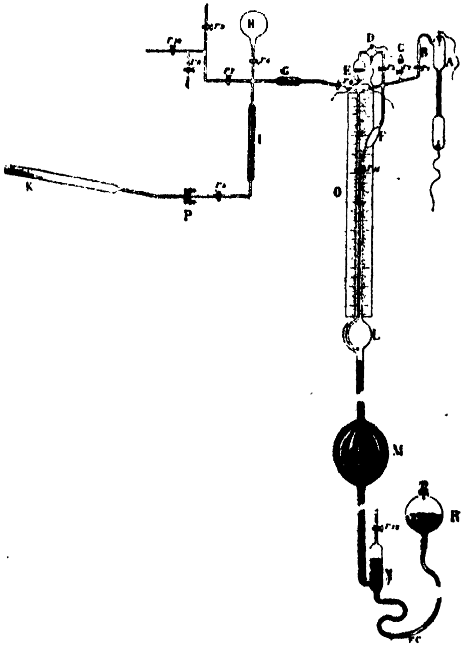
New Tannin Reaction and its Relation to the Anthocyanenes. K. Peche. (*Ber. d. Botan. Ges.*, 1914, **31**, 462-471; through *Chem. Zentralbl.*, 1914, II., 270.)—If sections of plants containing tannins that give a green coloration with iron (*e.g.*, the leaves or bark of *Prunus laurocerasus*) be heated with a mixture in equal parts of 20 per cent. potassium hydroxide solution and formaldehyde, a bluish-green colouring matter is formed in the cells containing the tannin. These coloured products are changed to red by acids, and become bluish-green again with alkali, and blue with ammonia. Except as regards solubility, they behave like the anthocyanenes. A soluble pigment of dark red colour forming bisulphite compounds, with an odour of vanilla, may be isolated from the sap of *Mespilus germanica.* The formaldehyde does not enter into the chromogenic reaction, but only protects the phenolic hydroxyl from oxidation. The pigments thus formed in rosaceous plants were derived solely from iron-greening tannins, and corresponded in locality with the natural anthocyanenes. In the case of *rosaceæ*, at all events, the latter are derived from the same group of tannoid substances. C. A. M.

Estimation of Urea. A. Desgrez and R. Moore. (*Comptes rend.*, 1914, **159**, 250-253.)—The decomposition of urea by Millon's reagent is greatly accelerated by the presence of infusorial earth as a catalytic agent, and is then quantitative at 17° C. The reagent is prepared by dissolving 50 grms. of mercury in 100 grms. of cold nitric acid, and adding to the solution an equal volume of the turbid liquid obtained by shaking 1 grm. of infusorial earth with 100 c.c. of water, allowing the mixture to stand for one minute, and decanting the supernatant liquid. A special ureometer, the lower part of which is curved upwards and ends in a bulb, is used for the estimation. The graduated body of the instrument is filled with chloroform by means of a capillary funnel passed through the stopper at the top. The urea solution, washing-water, and reagent are then successively introduced, and the ureometer immersed in water at about 25° C. In the case of urine 1 c.c. is used, with about 2 c.c. of water and 6 c.c. of the reagent. The last traces of carbon dioxide in the liberated gas are removed by introducing 2 c.c. of potassium hydroxide solution. The residual gas is measured in the usual way. The estimation is finished in forty minutes, while at 17° C. an hour is required. The only other nitrogenous constituent in urine decomposed by the reagent under these conditions is allantoin, but the amount of this excreted in human urine is very small—about 0·015 grm. in twenty-four hours. C. A. M.

INORGANIC ANALYSIS.

Detection of Bromides in Presence of Thiocyanates and Ferrocyanides: Action of Chromic Acid on Cyanogen Bromide. I. Guareschi. (*Zeitsch. anal. Chem.*, 1914, **53**, 490-495.)—The author has previously described a very sensitive reaction for bromine, depending on its liberation from its compounds by means of chromic acid and its subsequent action in restoring the colour to fuchsin which has been decolorised by treatment with sulphurous acid (ANALYST, 1913, **38**, 118, 518). This fails in presence of thiocyanates, if comparatively dilute chromic acid solution is added to the solution of bromide and thiocyanate. Under certain conditions, however, the test will detect 0·5 mgrm. of bromide in presence of 1 grm. of thiocyanate. For example, if 10 c.c. of 50 per cent. chromic acid be added slowly (on account of the violent reaction) to a mixture of solid potassium thiocyanate containing a trace of potassium bromide, and the mixture heated to incipient boiling, and a current of air then passed through it, sufficient bromine will accompany the air to affect a strip of paper treated with the fuchsin reagent. In the case of ammonium salts, the reaction with 50 per cent., or even 25 per cent., chromic acid is too violent, and it is better to use a large excess of 5 to 10 per cent. acid. Directions are given for the detection of traces of bromide in presence of the thiocyanates of silver and mercury and in presence of very large quantities of ferrocyanides. The test as originally described suffices when the ferrocyanide-bromide ratio does not exceed 1000 : 1. Cyanogen bromide itself is readily decomposed by chromic acid with liberation of bromine, the reaction being almost instantaneous with chromic acid concentrations above 15 per cent. With weaker acid it proceeds much more slowly, but a concentration as low as 0·2 per cent. suffices to bring it about.

G. C. J.



Analysis of Very Small Quantities of Gas: Application to Air Analysis.

P. A. Guye and F. E. E. Germann.

(*Comptes rend.*, 1914, **159**, 154-157.)—The principle of the method is the measurement of the pressure at which the sample (0·25 c.c. or less at *N. T. P.*) fills an apparatus of some 25 c.c. capacity, and the redetermination of this pressure after the sample has been subjected to the action of appropriate absorbents, etc. The method was first devised for studying the gases occluded in rocks, and the apparatus *K* represents a tube in which such rock can be heated and subjected to reduced pressure by means of the pump *R*, *N*, *M*. The dimensions of *K*, *I*, *G*, and *L*, are such that the space from r_4 to a mark below *L* is just equal to the space to the left of r_4. The gas to the right of r_4, half that in the sample, is usually taken for analysis, although nearly the whole could, if necessary, be drawn over by means of the pump. The initial pressure, preferably about 7 to 8 mm., is read, like subsequent pressures, to within 0·01 mm. by means of the MacLeod gauge O, L, r_{11}. The manner in which the gas can be brought into contact with appropriate absorbents, etc., in A, B, C, D, etc., is clear from the drawing. For the authors' purposes, E contained an iron spiral, with electrical connections for raising it to such a temperature as is requisite to make it function as an absorber of oxygen. C contained potash, D platinum electrodes for sparking with oxygen or hydrogen, and B gold leaf to absorb mercury vapour, which would otherwise attack the aluminium electrodes of the Plücker tube A, which serves for the spectroscopic examination of the gas residue. It is suggested that the apparatus would be useful for the analysis of air from high altitudes of which it is difficult to obtain large samples. G. C. J.

Commercial Copper Sulphate. F. Mach and P. Lederle. (*Landw. Vers.-Stat.*, 1914, **84**, 129-143; through *Chem. Zentralbl.*, 1914, II., 352-353.)—In all cases the sum of the constituents amounted to somewhat more than 100 per cent.; possibly the fact that the copper was estimated electrolytically introduced a slight error. One sample examined contained 1·2 per cent. of manganese sulphate. Powdered copper sulphates examined not unfrequently contained considerable quantities (12 to 15 per cent.) of ferrous sulphate. The method described by Windisch (ANALYST, 1913, **38**, 75; see also p. 520) is suitable for the rapid estimation of copper, but tends to yield low results. Moser's method of estimating copper (ANALYST, 1912, **37**, 516) by titration with titanous chloride is trustworthy.

W. P. S.

Gas Analysis by Fractional Distillation at Low Temperatures. G. A. Burrell and F. M. Seibert. (*J. Amer. Chem. Soc.*, 1914, **36**, 1537-1548.)—A development of the method of Lebeau and Damiens (ANALYST, 1913, **38**, 121, 276). These authors obtained three fractions, one containing the whole of the hydrogen and methane and nothing else, another containing the whole of the ethane with some propane, and the third containing the rest of the propane with any butanes. The eudiometric analysis of these mixtures of only two gases was then easy. The present authors make a sharp separation of methane, ethane, and propane by fractional distillation, analysis of the separated gases by slow combustion showing that they were of 99·9 per cent. purity, and that the residual fraction in the case of natural gas was mainly butane. Methane is distilled at $-185°$ C. (63 mm.). The distillate is always pure methane, but the residue, on being allowed to attain the gaseous state, then being liquefied and again distilled at $-185°$ C. (63 mm.), usually yields about 1 per cent. more methane. The chief separation of ethane is effected as follows: The liquid is surrounded by a bath of "wild" gasoline, cooled by means of liquid air to $-140°$ C. "Wild" gasoline is the condensate obtained from some natural gases by compressing them to 250 lbs./in.^{2}, and then cooling to air temperature, and remains quite fluid at low temperatures, unlike ether, etc. With the apparatus described and illustrated in the paper, the temperature of the "wild" gasoline bath rises about 5° C. to 10° C. per hour, after the surrounding liquid air has all evaporated, and it is allowed to rise to $-125°$ C., whilst the gas to be analysed is gradually deprived of nearly all its ethane and some propane by means of the pump, the pressure finally falling to about 3 mm. Both distillate and residue are refractionated, the former after liquefaction, the latter after being allowed to attain the gaseous state and being reliquefied. The combined distillates now contain the whole of the ethane, but require to be fractionated twice more, at −155° to −140° C. and at −145° C., to eliminate the last of the propane. A still greater number of fractionations is necessary to obtain the propane free from butane, the temperature and pressure of separation ranging from −135° C. at 0·1 mm. to −110° C. at 1 mm.

G. C. J.

Permanganate Titration of Iron in Presence of Chlorides. O. L. Barnebey. (*J. Amer. Chem. Soc.*, 1914, **36**, 1429-1448.)—The author finds that the Zimmerman-Reinhardt method gives exact results, and suggests that the small constant error found by Jones and Jeffery (ANALYST, 1909, **34**, 306) was due to a trace of iron in their stannous chloride. When titrating small amounts of iron in a volume of 400 to 600 c.c., the use of phosphoric acid, as directed by Reinhardt, is unnecessary, provided sufficient sulphuric acid and manganese are present. In general, the disturbing action of hydrochloric acid is inhibited if the volume of Reinhardt solution taken is equal to the volume of hydrochloric acid (sp. gr. 1·1) present. These were the proportions recommended by Jones and Jeffery (*loc. cit.*), and the statement of these authors is confirmed that the use of an unnecessary excess of stannous chloride, with subsequent formation of a notable amount of mercurous chloride, tends to give high results. Certain neutral sulphates and acid phosphate mixtures react with hydrochloric acid and prevent the latter from causing high results in the iron titration, acid phosphate solutions being especially serviceable, and such mixtures may replace Reinhardt's solution. The author discusses the various theories that have been brought forward to explain the function of manganese salts in the titration of iron with permanganate, citing some thirty papers, and expresses the opinion that Birch's view (*Chem. News*, 1909, **99**, 61; ANALYST, 1909, **34**, 315), that $MnCl_3$ is formed, is deserving of more general consideration than has been accorded it by subsequent authors. G. C. J.

Note by Abstractor.—The results of Jones and Jeffery cannot be explained by the presence of iron in their stannous chloride, as this would not have introduced a constant error, widely varying amounts of stannous chloride having been used.

Analysis of Pyrophoric Alloys of Cerium and Iron. H. Arnold. (*Zeitsch. anal. Chem.*, 1914, **53**, 496-503.)—In the analysis of these alloys it has been usual to separate the cerium earths first as oxalates, and then, after destruction of oxalic acid, to precipitate iron with ammonia. Often the percentage of iron is the figure most needed or the only figure required, as many of these alloys contain little besides iron and metals of the cerium group (Ce, La, Di, etc.), and as the sole object of analysis is sometimes to determine whether or not the alloy infringes Auer's patent for alloys of cerium metals with 30 per cent. or more of iron. The above method is slow, as the separation of the cerium metals as oxalates is not even approximately quantitative unless twelve hours or more be allowed before filtration, and the subsequent iron oxide precipitate is invariably contaminated with cerium earths, the final separation of which is a very tedious operation.

The author has tried various methods for the direct determination of iron, to be followed, if necessary, by the estimation of the other constituents of the alloy. He recommends precipitation of iron as sulphide by addition of ammonia and ammonium sulphide to a tartaric acid solution. The alloy is covered with water and dissolved by means of hydrochloric acid and bromine, the latter preventing loss of silicon as hydride. Silica is separated in the usual manner, a little potassium chloride being added before evaporation to prevent volatilisation of antimony chloride during baking. The filtrate from the silica has 3 to 5 grms. tartaric acid added to it, and is then poured into 50 c.c. of strong ammonia and heated to about 60° C. Ammonium sulphide solution (15 to 30 c.c.) is added slowly, with constant stirring, and the mixture is kept covered on the water-bath for one and a half to two hours. The precipitate, which often contains notable amounts of copper, and occasionally some zinc as well as iron, is filtered through a dense filter, with the usual precautions against oxidation, and washed some fifteen times with a warm solution of ammonium sulphide containing ammonia and ammonium tartrate. If zinc is present, the filter must be treated with nitric acid before ignition to avoid loss by volatilisation. If the percentage of iron alone is required, the precipitate is dissolved in hydrochloric acid, with addition of potassium chlorate, and the iron estimated volumetrically. As copper up to 5 per cent. is present in some alloys, however, it will often be necessary to ignite and dissolve the precipitate in hydrochloric acid, and separate copper as sulphide in acid solution. Iron is then separated from any zinc in the usual manner. If manganese is present, the iron in the sulphide precipitate is best separated from accompanying metals by an ether separation of the chlorides. The filtrate from the sulphides should throw no further precipitate on standing one to two hours. If left overnight, it should have more ammonia added to it, and be covered to prevent any ambiguity due to separation of sulphur. The tartaric acid in the filtrate is best destroyed by heating the concentrated solution for an hour on the sandbath, with 10 c.c. nitric acid and 2 grms. potassium chlorate. The mixture is then evaporated to dryness, the residue taken up with a further 10 c.c. of acid, and, after addition of 1 grm. potassium chlorate, heated on the sandbath another fifteen to thirty minutes. The nearly dry residue is largely diluted, and the cerium earths precipitated by means of oxalic acid, and filtered off after twelve hours. In the filtrate, oxalic acid is destroyed by means of nitric acid, and any antimony precipitated as sulphide. In the filtrate from the antimony, aluminium is precipitated by means of ammonia.

G. C. J.

Determination of Iron in Presence of Phosphoric Acid. C. E. Corfield and W. R. Pratt. (*Brit. Pharm. Conference*, 1914, 19.)—Attempts to evolve an accurate gravimetric method for the determination of iron in presence of phosphoric acid failed. In investigating volumetric methods it was found that phosphoric acid retards the reduction of ferric salts and the liberation of iodine from hydriodic acid by ferric salts, rendering these methods tedious. Methods involving reduction by zinc are inaccurate apart from the presence of phosphoric acid, since the end-point is obscured by the zinc salts; in dilute solution the blue ferrous ferricyanide is prevented from forming, and in concentrated solutions is obscured by the zinc ferricyanide. Reduction by amalgamated aluminium, tin, and stannous chloride gave good results. The stannous chloride method is recommended. O. E. M.

Manganese Sulphide and the Estimation of Manganese. A. Villiers. (*Comptes rend.*, 1914, **159**, 67-70.)—The dense green form of manganese sulphide is most easy to filter and wash. The conditions requisite to insure precipitation of manganese in this state appear to differ from those which insure the formation of other metallic sulphides in a dense, easily filtered form. This is said to be due to the fact that the pink sulphide exists in at least two states, the least dense of which is readily converted into the green, crystalline sulphide, whilst the denser pink sulphide is quite stable even at 100° C. The directions usually given for precipitating manganese as the green sulphide are to make the precipitation at a temperature near 100° C., and to add ammonia after adding ammonium sulphide. The author finds that under certain conditions the precipitation may be made in the cold and without the presence of excess of ammonia, but when ammonia must be added—and this will usually be the case in ordinary analytical practice—it should be added before the ammonium sulphide. The green sulphide can always be obtained by making the solution strongly ammoniacal, heating to 100° C., and then adding ammonium sulphide. The transformation is complete in seven to eight minutes. In absence of other salts, such as ammonium salts resulting from the neutralisation of acids, or in presence of very large quantities of such salts, the precipitation may be effected in neutral solution. In absence of ammonia, however, moderate saline concentrations induce the formation of the stable pink sulphide, which cannot be transformed into the green modification by subsequent addition of ammonia and boiling. In very dilute, neutral solutions, free from foreign salts, the green sulphide may be obtained even at room temperature. The precipitate is then grey at first, becoming greenish in ten minutes, and quite green in twenty minutes, the transformation being complete in forty-five minutes. G. C. J.

Nickel Cooking-Vessels. M. Vuk. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1914, **28**, 103-104.)—In view of contradictory statements as to the liability of nickel vessels to attack food substances by acid, the author has carried out a number of experiments, and finds the degree to which the vessels are attacked by dilute acetic acid depends on the character of the nickel used in making the vessels. The following quantities per sq. dm. were dissolved when different grades of nickel plate were heated on a boiling water-bath with 5 per cent. acetic acid for two and a half hours; rolled nickel, 16 mgrms.; cast nickel, 28 mgrms.; electrolytic nickel, 31 mgrms.; drawn nickel, 39 mgrms.; "pure" nickel, 65 mgrms.

W. P. S.

Volumetric Estimation of Small Quantities of Potash. L. Zaleski. (*Landw. Vers.-Stat.*, 1913, **83**, 221-255; through *Chem. Zentralbl.*, 1914, I., 2121.)—The author has tested the method described by Mitscherlich (ANALYST, 1912, **37**, 588). Since in this process the evaporation of the liquid containing the potassium cobaltinitrite is objectionable, he has endeavoured to modify the method in such a way as to precipitate the potassium nitrite directly in the solution in presence of nitric acid with the reagent recommended by de Koninck (ANALYST, 1909, **34**, 114) for the detection of potassium. In order to obtain the best results possible by this modified method, the precipitation must be effected by a considerable excess of the reagent (0·5 to 1·0 c.c. for each 1 mgrm. of K_2O). The necessary quantity of the reagent is added slowly while stirring, and the precipitate filtered off after twelve to twenty-four hours. The potash-factor is determined at the same time and under the same conditions. Neither Mitscherlich's method nor the modification now described is quite accurate. By close adherence to all necessary conditions, however, very satisfactory results can be obtained by both methods. The Mitscherlich method presents certain difficulties, and the author's modification affords a simpler means for arriving at results of the same degree of accuracy. J. F. B.

Quantitative Determination of Radium Emanation in the Atmosphere, and its Variation with Altitude and Meteorological Conditions. J. R. Wright and O. F. Smith. (*Philippine J. of Science*, 1914, 9 A, 53-76.)—The method employed was essentially identical with that of Eve and Satterly (*Phil. Mag.*, 1907, **14**, 724; 1908, **16**, 584, 622), large quantities of air being drawn over coconut charcoal contained in a tube 100 cm. long and 1·5 cm. in diameter. Rutherford (*Nature*, 1906, **74**, 634) showed that this was a good absorbent for radium emanation, and it is now shown that, for quantities of the order of magnitude dealt with by the authors, the absorption is quantitative. When the charcoal is distributed as a short column of greater diameter, a phenomenon analogous to saturation manifests itself, and with much higher concentrations of the emanation this phenomenon might be expected to manifest itself even when a long column of charcoal is used. At the same time air is bubbled through a solution of radium bromide containing a known amount of radium ($6·3 \times 10^{-10}$ grm.), and the emanation from the solution and the air is collected in another charcoal tube. The emanation absorbed in the charcoal is then driven off by heating to a dull red heat, collected over water in aspirators, and finally measured by passing into an ionisation chamber connected either with an electroscope or an electrometer. The emanation in a given volume of air can then be calculated from the ratio—

$$\frac{\text{emanation generated in the standard solution in a known time}}{\text{emanation in a known volume of air}},$$

provided the charcoal tubes absorb the same fraction of the total amount of emanation passing through them. This point was left in doubt by earlier workers, but the present authors show that absorption, under their conditions, is substantially complete. On the other hand, bubbling air through a cold solution of radium bromide does not remove the emanation as rapidly as it is formed, as was assumed by both Satterly and Eve. Only about 80 per cent. of the emanation is removed at a temperature of 27° C.

Other workers, in different parts of the world, have reported radium emanation equivalent to 60 to 100 × 10^{-12} grm. radium per cubic metre of air. The mean value in Manila is about 80 × 10^{-12}, but the diurnal variations are large, extreme values being about 40 and 160 × 10^{-12}. On Mount Panui, at an elevation of about 8,000 feet, the mean value was about 20 × 10^{-12}, and the extreme values 10 and 40 × 10^{-12}. This suggests that the concentration of radium emanation in the atmosphere decreases as the altitude increases. The lower values were found during or shortly after rain had fallen. Night values are, on the average, twice as high as day values. No definite relation could be traced between the variation of emanation content and a rising or falling barometer. Changes in humidity, likewise, seem to have no effect on the radio-activity of the atmosphere, but the total wind movement is evidently an important factor in determining the variation. G. C. J.

Detection of Small Quantities of Selenious Acid in Sulphuric Acid. E. Schmidt. (*Arch. Pharm.*, 1914, **252**, 161-165.)—On shaking 10 c.c. of sulphuric acid with 0·01 grm. of codeine phosphate, a green coloration, changing to bluish-green in fifteen minutes, is obtained in the presence of traces of selenious acid (0·00005 grm.). The test is thus more sensitive than the sodium hydrosulphite test (red coloration due to separated selenium). Sulphuric acid containing sulphurous acid gives no coloration with codeine phosphate, while the characteristic coloration is produced on adding 0·0001 per cent. of selenious acid. Tellurous acid at first does not affect sulphuric acid containing codeine phosphate, but after some time a red or pale blue colour is produced. On now adding selenious acid the distinctive green coloration is obtained, though more slowly than in the absence of tellurium.

C. A. M.

Volumetric Estimation of Titanium and Chromium by Means of a Modified Reductor. C. Van Brunt. (*J. Amer. Chem. Soc.*, 1914, **36**, 1426-1429.) —Shimer and Shimer's method for titanium (ANALYST, 1912, **37**, 590) is slow, especially when the only zinc available is finer than was specified by these authors. Moreover, the 3′ reductor is inconvenient, and it is difficult to prevent reoxidation.

The author uses an ordinary Jones reductor with a column of amalgamated zinc about 8″ high and 1″ in diameter. The tube is wrapped with a few feet of resistance ribbon or iron wire, in series with a lamp or other resistance adapted to taking sufficient current from a lighting circuit to boil the solution in the reductor. With this device, not only titanic, but also chromic salts, can be reduced in a few minutes. The stopcock at the bottom is closed, and the tube partly filled with hot 5 per cent. sulphuric acid, which is followed by the solution to be reduced. The apparatus is closed at the top by means of a double-bored stopper, carrying a tap-funnel and a vent-tube. The solution is kept near the boiling-point for ten to twenty minutes, and the pressure of steam is then made to expel the reduced solution through the stopcock at the bottom and below the surface of a ferric solution. The resulting ferrous salt, being comparatively stable, does not need to be protected against access of air, as does a titanous solution. The zinc in the reductor is washed successively with 5 per cent. and 1 per cent. sulphuric acid, which are admitted by means of the tap funnel and expelled by steam pressure without access of air, and also collected below the ferric solution. The ferrous salt is then titrated with permanganate.

Chromic salts may be reduced to chromous salts in a similar manner, which are then caused to react with ferric salts, and the resulting ferrous salt estimated as described. The application of the method to the estimation of iron, titanium, and chromium, when present together, is discussed. For the estimation of all three metals together, the procedure is as described above. Iron alone is estimated by passing the solution through a reductor in the ordinary manner, not collecting in ferric solution, but adding bismuth oxide to reoxidise any reduced titanium or chromium, or, the iron is reduced with hydrogen sulphide or sulphur dioxide, which do not reduce titanium or chromium. Chromium alone is estimated by oxidising to chromate with ammonium persulphate and titration in the usual manner with ferrous iron and permanganate. Titanium is then estimated by difference.

G. C. J.

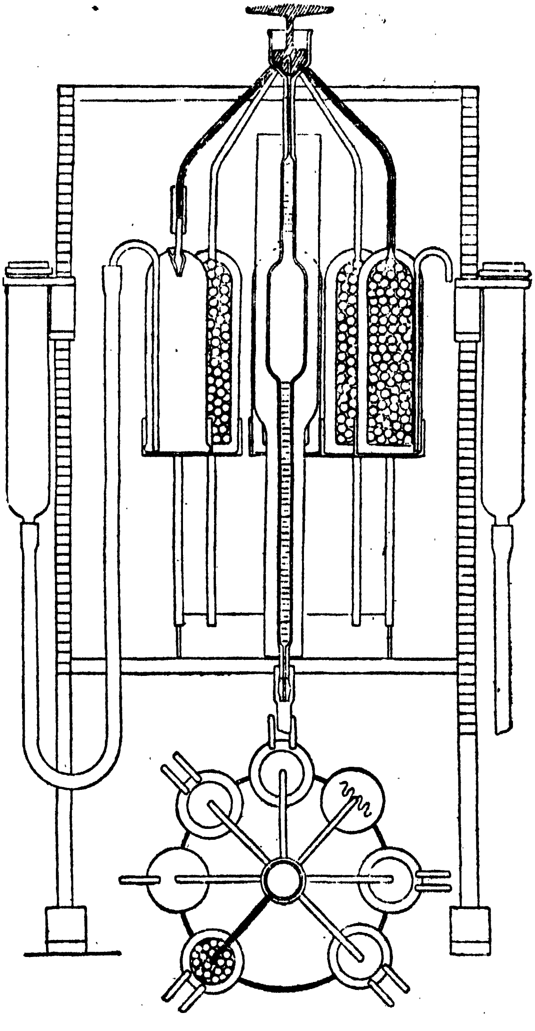
Separation of Yttrium from the Yttrium Earths. Part II. H. C. Holden and C. James. (*J. Amer. Chem. Soc.*, 1914, **36**, 1418-1423.)—In addition to the methods previously investigated (ANALYST, 1914, 280), the authors have tried fractional precipitation with sodium nitrite as well as fractionation by boiling with sodium hydroxide. Fractional precipitation with sodium nitrite is the most efficient and rapid method found thus far, the yttrium being concentrated in the mother liquors. This method is also less costly than either the phosphate or chromate methods formerly described (*loc. cit.*), and seems well adapted for the separation of yttrium on a commercial scale. It will not separate yttrium from terbium, since this element also tends to become concentrated in the mother liquors. G. C. J.

APPARATUS, ETC.

Apparatus for determining Boiling-Points without Thermometer Corrections. T. Paul and K. Schantz. (*Ber.*, 1914, **47**, 2285.)—The liquid is boiled in a large test-tube containing beads to secure even boiling, and standing over a hole, closed by wire gauze, in an asbestos plate. The vapour rises through a tube, concentric with the thermometer and enclosed in a jacket, and descends in the annular space between tube and jacket to a side-tube connected with a condenser. The condenser has a bent neck, and by rotating it through 180° can be made to act either as an ordinary or as a reflux condenser, so that distillations may be carried out, or the liquid returned to the jacket, and thence, by a trapped side-tube, to the boiling liquid.

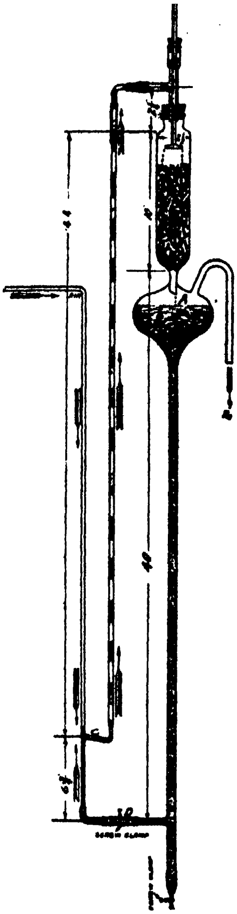
O. E. M.

Extraction of Finely Powdered Substances. E. Bornemann. (*Chem. Zeit.*, 1914, **38**, 833.)—A short tube of filtering material is fitted in the axis of an extraction thimble, through the bottom of which it passes tightly; the substance to be extracted is placed in the annular space thus enclosed. The arrangement is best used in a modified Soxhlet apparatus having no siphon, but an opening, through which the filtering-tube projects downwards, in the bottom of the extraction chamber, the walls of which have projections to centre the thimble. A glass bulb, to distribute the extracting liquid, lies mouth downwards on the open end of the thimble, which it exceeds somewhat in diameter; the upper end of the filtering-tube, closed with cotton-wool, projects into its interior. It is pierced with one hole in a slight depression at its highest point, and a ring of holes above its equator. Resting on it is a glass ring, to promote the distribution of the solvent. The apparatus, thimbles, and tubes, may be obtained from Machery, Nagel and Co., of Düren, or of the Ver. Fabriken für Laboratoriumsbedarf, Berlin. O. E. M.



Gas Analysis Apparatus. G. Wempe. (*Chem. Zeit.*, 1914, **38**, 794-795.)—The illustration shows a longitudinal section through the measuring-tube of a new apparatus for technical gas analysis designed to do away with stopcocks and rubber connections as far as possible. In the lower part of the figure the arrangement of the absorption pipettes is shown in plan. The central measuring-tube has a capacity of about 60 c.c., and is enlarged near its upper end in the form of a cylindrical bulb. Both ends are extended in a capillary bore, the zero mark being on the top capillary and 1000 on the bottom. The upper portion above the bulb carries graduations from 0 to 70, and the portion below the bulb continues the graduations from 770 to 1000. At the top, just above the zero mark, is a stopcock of the form shown, ground into a cup which may be made gas-tight by means of mercury. This stopcock may be turned so as to make communication between the measuring-tube and any one of a number of absorption vessels, arranged in the form of a circle, by way of capillary tubes. The absorption vessels consist of cylinders filled with glass balls and having perforated bottoms, each being enclosed in bell-jars by fused joints. A bent tube passes through to the bottom of each vessel, through which the reagent can be changed without dismounting the apparatus; this tube can be closed by a stopcock (not shown), so that the liquid will not move when the central tap is rotated. The sample of gas is introduced through a capillary branch which is not provided with an absorption vessel. Schorndorff's arrangement is provided for adjusting the zero-point and compensating differences of temperature between two readings.

J. F. B.



Nitrogen Generator. C. Van Brunt. (*J. Amer. Chem. Soc.*, 1914, **36**, 1448-1450.)—The apparatus figured is intended to deprive air of oxygen by means of copper and ammonium carbonate solution. The novelty consists in the continuous circulation of the solution through the copper column, *B*, by means of an air-lift actuated by the incoming air-current.

The larger dimensions are given in the drawing, which is nearly self-explanatory. The diameter of the riser tube should not exceed 5 to 6 mm. unless an unusually rapid current of gas is required. The reservoir *A* may be of any size, depending on the purpose to be served. It is charged with a mixture in equal proportions of a saturated solution of commercial "ammonium carbonate" and ammonia solution of sp. gr. 0·93. One litre of such a mixture serves for the preparation of 75 litres of "nitrogen" from air. In constructing the apparatus, care must be taken that the tube *C* is slightly inclined to the horizontal, as shown. This insures a steady operation of the lift. If the dimensions shown in the drawing are deviated from, it is necessary only to remember that the hydrostatic pressure of the liquid in the reservoir should overbalance that of the column of mixed gas and liquid in the riser by an amount equivalent to the required "velocity head." Adjustment of the relative proportions of air and solution is made by means of the screw clamp *D*. The "nitrogen" passing away from *A* is washed with dilute sulphuric acid contained in appropriate apparatus.

G. C. J.

Stirring Apparatus. W. Gluud and R. Kempf. (*J. Soc. Chem. Ind.*, 1914, **33**, 680.)—The apparatus consists of a glass vessel with three stoppers, the outer ones fitted with a reflux condenser and a dropping funnel, respectively, and the central one with a vertical shaft, provided at the bottom with cross-rods for stirring, and at the top with a band pulley secured by a split chuck. A seal, which may be filled with water, paraffin, mercury, etc., is placed below the stopper, and prevents the escape of vapour round the spindle. The vessel has a capacity of 1,500 c.c., but is constricted at the bottom so that 150 c.c. of liquid may be effectually stirred. The apparatus is sold by Gebr. Muencke, Schumannstr. 2, Berlin, N.W. O. E. M.

Fontactoscope for measuring the Radio-Activity of Spring Waters. C. Engler, H. Sieveking, and A. Koenig. (*Phys. Zeitsch.*, 1914, **15**, 441; through *Chem. Zentralbl.*, 1914, I., 2205.)—Investigations similar to those of Hammer and Vohsen (*Phys. Zeitsch.*, 1913, **14**, 451) were carried out with two fontactoscopes made by Engler and Sieveking; the results were found to be accurate to 1 or 2 per cent. The results were the same whether the instrument was left open or shut during a measurement lasting not more than one hour, or whether the water was left in the instrument after shaking, or run off. The activity curves obtained agreed well with the theoretical, especially for the first hour. The residual activity, after boiling out the water, was found to be 2 per cent. or less, and consequently negligible. It is thus possible to determine the radio-activity of such waters with the instrument to a sufficient degree of accuracy. O. E. M.

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REVIEW.

Principles and Practice of Agricultural Analysis. By H. W. Wiley. Chemical Publishing Company, Easton, Pa. 1914. Pp. 846. Price. $6.00.

The first edition of Wiley's "Agricultural Analysis" completely captured the agricultural world, and soon became perfectly indispensable both to the analyst and the agricultural chemist. Now a second edition is issued, and in going through it one cannot help wondering how the author has managed to keep it within reasonable compass, and whether it can possibly be better than the old edition.

The first thing that strikes the reviewer with pleasure is that the characteristic features of the book are retained, and, if Dr. Wiley's wishes are respected, they will be retained in future issues. "Subsequent editions of this work will probably be undertaken by other hands than mine. I simply would urge upon the revisers of the future to retain, if possible, the cardinal principle on which this work is based—namely, to present in as compact a form as possible a connected, historical story of the development of agricultural analysis, emphasising the fundamental principles on which it is based." This "historical story" is interesting in any branch of science, but it is indispensable in agricultural analysis for a very important reason.

Agricultural analysis is not usually absolute; there is commonly a convention or two lurking somewhere in the method, and the analytical results have value only for purposes of comparison. Thus, in the simple case of soil analysis: the so-called "total" potash is not the total at all, but only the amount extracted by an acid under certain conditions, and is different from the result obtained when another acid is used or other conditions substituted. Of course, if all analysts agree on a method, it may prove very useful for instituting comparisons—often in agricultural work the chief purpose of the work—and in each country a certain amount of agreement is, in point of fact, attained. But there is no international agreement, and widely different methods are in use in different countries; the same terms are used, but they stand for different things. The great merit of Dr. Wiley's book is that it gives sufficiently full accounts of the chief methods to enable investigators and analysts in any one country to follow the work of their colleagues elsewhere, and reference to its pages often enables one to clear up apparent discrepancies.

The present volume—the third—deals with agricultural products, and includes sugars and starches, fats and oils, proteins, etc., dairy products, and various fodders. Special attention is given to methods of preparation of the samples and similar preliminary processes, which play so conspicuous a part in determining the success or failure of the analysis, and then the detailed operations are given. All this is condensed into a little over 800 pages, so that no space was available for superfluities, and the reader who expects to find a complete summary of all known analytical processes will be doomed to disappointment. It is obviously impossible in any one laboratory to test all the methods that are proposed, and Dr. Wiley seems to have adopted the rather drastic plan of selecting only those that have survived for several years, without attempting to discriminate between the many newer methods that are daily cast on the waters, to sink or swim as their own merits shall determine. This plan has the advantage of maintaining some sort of perspective, but it has the disadvantage that it does not make adequate use of numerous papers published during the last four or five years.

Take, for example, the determination of sugar and starch in leaves, grains, roots, etc. The subject is of considerable importance, and is likely to become more so in the future as the chemist is expected to correlate the quality of crops with their manurial treatment. The current methods have been carefully and systematically tested by Mr. Davis at Rothamsted, and they have been found defective; modifications and new methods have been introduced which give considerably better results than the old ones. An account of this work was published in 1913, yet no reference occurs in the book. Again, Wein's tables for use in maltose determinations are given at length, although Brown and Morris have shown that they break down entirely when other sugars are present, and are liable to an error of 5 per cent. even in dealing with pure maltose. So, again, no mention is made of Pellet's paper on the sources of error in the ordinary inversion process. Instances might be multiplied, but no doubt Dr. Wiley is waiting to see how all this 1912 and 1913 work is going to survive before deciding just what to put in and what to omit. There is much to be said for this plan, but we think that new work of so high an order of merit as that of Davis and Daish ought to have been included.

The historical part is much as it was, which is the same as saying that it is about as good as it could be. We hope, however, that in a future edition the history of mutarotation will be put right. This phenomenon has, of course, been known for a long time, but it was not satisfactorily cleared up till 1903, and the credit of putting it on a sound scientific basis is due to Lowry. Yet Lowry's paper is not mentioned.

However, the analyst who gets hold of Dr. Wiley's book will feel that he has much to be thankful for, and he will not be disposed to quarrel because of the omissions. Our purpose in pointing out these half-dozen instances is to urge that they should be noted for a future edition. The book is so good in itself that its revisers cannot afford to overlook good work done elsewhere on the subject.

E. J. RUSSELL.

OCTOBER, 1914. Vol. XXXIX., No. 463.

THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

THE CHIEF CAUSE OF THE LOSS OF SULPHURIC ANHYDRIDE AND OF CHLORINE BY INCINERATING SUBSTANCES CONTAINING THESE CONSTITUENTS.

By JAMES O'SULLIVAN, F.I.C.

(*Read at the Meeting, June* 3, 1914.)

Thudicum and Dupré, "Origin, Nature, and Varieties of Wine," 1872, referred to the loss of sulphuric anhydride and chlorine when the residues from certain wines were incinerated. At that time analysts who had experience in the estimation of chlorine and sulphuric anhydride in beer were aware that the quantity of either of these found in the ash of the beer was not a correct measure. I also know that analysts from time to time have fallen into this analytical trap, and certified beer to contain "chlorine traces" and an amount of sulphuric anhydride which was practically only one-half of what was present.

Ratcliff (Analyst, 1907, **32**, 84) drew attention to the discrepancy between the sulphuric anhydride found in vinegar by direct precipitation, and the amount found in the ash, and he states "that this difference cannot be satisfactorily explained on the assumption that the carbonaceous matter reduces the sulphates to sulphides"; he inferred that the phosphate present expels the sulphuric anhydride on ignition. Ratcliff also knew that in the estimation of chlorine it was necessary to add sodium carbonate before charring, and that the sulphuric anhydride should be precipitated direct.

The sulphuric anhydride was estimated by direct precipitation in a genuine hock, which by the Haas method showed no sulphurous acid, in grape wine made with Burton water, and in vinegar, and also in the ash of these, with the following results:

	Grm. per 100 c.c.		
	Hock.	Grape-Wine.	Vinegar.
Direct	0·060	0·089	0·125
Ash	0·062	0·038	0·107
Loss	none	0·051	0·018

The ash of the hock amounted to 0·281 grm.; it contained carbonate, and its alkalinity was equal to 0·164 grm. K_2O; the ash of the grape wine and vinegar was alkaline, and contained magnesium oxide, but no carbonate or chloride.

Table I. gives the results in grm. of sulphuric anhydride per 100 c.c. of beer and stout.

TABLE I.

	Beer.				Stout.			
	a.	*b.*	*c.*	*d.*	*a.*	*b.*	*c.*	*d.*
Original gravity ...	1062°	1061°	1061°	1082°	1077°	1076°	1079°	1076°.
SO_3 found by direct precipitation ...	0·130	0·127	0·124	0·124	0·097	0·116	0·114	0·114
In ash	0·080	0·069	0·078	0·074	0·034	0·055	0·063	0·063
Loss	0·050	0·058	0·046	0·050	0·063	0·061	0·051	0·051

The chlorine was determined in the ash obtained from Beer *d* and Stout *d* in the ordinary way, and also by evaporating 100 c.c. with 10 grms. of lime and incinerating the residue.

	Grm. per 100 c.c.	
	Beer *d.*	Stout *d.*
Incinerated in presence of CaO ...	0·026	0·026
Without CaO	nil	nil
Loss	0·026	0·026

The mineral residues of these beers and stouts contained magnesium oxide, but no carbonate or sulphide.

The alkalinity of the ash from Beer *d* was equal to 0·055 grm. of magnesium oxide, and in Stout *d* to 0·059 grm., and the amount of magnesium oxide found in the ash was 0·048 grm. and 0·047 grm. respectively.

The alkalinity was determined by adding an excess of $\frac{N}{10}$ HCl to the ash, filtering, and titrating the filtrate with $\frac{N}{10}$ potash, phenolphthaleïn being used as indicator. If the ash of beer or stout is extracted with water and filtered, only about one-tenth of its total alkalinity will be found in the filtrate. It is obvious that in these beers and stouts the amount of chlorine and sulphuric anhydride in the ash is not a measure of the quantities present.

The sulphates present in beer and stout are calcium, magnesium, and chiefly potassium sulphate, and the question arises, which of these sulphates is decomposed, on incinerating, to an extent sufficient to account for the great loss of sulphuric anhydride and chlorine.

Fresenius ("Quantitative Analysis," sixth edition, 1873, p. 745) pointed out "that sulphate of magnesium when exposed in a small platinum crucible for fifteen to twenty minutes to the heat of a powerful blast gas-lamp gave, with dilute hydrochloric acid, a solution in which barium chloride failed to produce the least turbidity." That the destruction of sulphate of magnesium explains the loss

USSELI

chief of the chlorine and of the sulphuric anhydride on incinerating substances containing that salt, the following experiments will show.

Estimations were made by direct precipitation in solutions of different sulphates, and also in the ash from the same quantity of the sulphates, previously mixed with 2 grms. of cane-sugar.

TABLE II.

	(1) $CaSO_4$ Solution. SO_3 Grm.	(2) K_2SO_4 0·201 Grm. SO_3 Grm.	(3) $MgSO_4 7H_2O$ 0·255 Grm. SO_3 Grm.	(4) $MgSO_4 7H_2O$ 0·255 Grm. SO_3 Grm.	(5) Na_2SO_4 0·204 Grm. SO_3 Grm.
Direct precipitation	0·112	0·096	0·082	0·082	0·117
In ash	0·108	0·095	0·009	0·008	0·110
Loss	0·004	0·001	0·073	0·074	0·007
Loss per cent. ...	3·5	1·4	89·0	90·2	5·9

The weight of ash in Experiment 3, Table II., was 0·054 grm.; its alkalinity, calculated as MgO, amounted to 0·0365 grm.; and the quantity of sulphuric anhydride in the ash, calculated as $MgSO_4$, amounted to 0·0134 grm., or 0·0365 MgO + 0·0134 $MgSO_4$ = 0·0499 grm., against 0·054 grm. weighed.

Solutions of the sulphates of calcium and magnesium were each precipitated with phosphate of potassium; the precipitates were dissolved in acetic acid and made up to 100 c.c. In one half of each solution the sulphuric anhydride was determined direct, to the other half 2 grms. of cane-sugar were added, and the residues, after evaporation, incinerated. The sulphuric anhydride was also determined in a mixture of sulphate and phosphate of potassium, and in a similar mixture after subjecting it to the same conditions as in an ash determination. The following results were obtained:

TABLE III.

	(1) $CaSO_4$. SO_3 Grm.	(2) $MgSO_4 7H_2O$. SO_3 Grm.	(3) $MgSO_4 7H_2O$ 0·393 Grm. SO_3 Grm.	(4) K_2SO_4 0·2 Grm. K_3PO_4 0·24 Grm. SO_3 Grm.
Direct precipitation	0·010	0·064	0·126	0·092
In ash	0·009	0·025	0·037	0·092
Loss	0·001	0·039	0·089	nil
Loss per cent. ...	10·0	60·9	70·6	nil

If free phosphoric acid is present in a solution containing sulphate, and it is desired to estimate the sulphuric anhydride, the barium precipitate must be repeatedly washed with boiling dilute hydrochloric acid to free it from phosphate. In my experience this is a difficult estimation, unless the phosphoric acid is first con- into a phosphate.

TABLE IV.

Behaviour of Magnesium Sulphate and Potassium Chloride when Ignited.

	Experiment I. Ignited together.	Experiment II. Ignited in Presence of Cane-Sugar.	Experiment III. KCl ignited with Cane-Sugar.
SO_3 present per cent.	32·3	32·3	—
SO_3 found ,, ...	32·5	13·9	—
Cl present ,, ...	47·1	47·1	47·1
Cl found ,, ...	5·5	nil	28·4

It is clear from these experiments that the chlorine is lost on ignition with magnesium sulphate, that the sulphuric anhydride is fixed by the potassium, and that in the presence of organic matter (Experiment II.) both sulphuric anhydride and chlorine are lost.

The decomposition of magnesium sulphate on ignition being a question of time and temperature, I have little doubt that had the ignition in Experiment I. been prolonged the whole of the chlorine would have been displaced by the sulphuric anhydride, and the quantity of the latter in excess of what the potassium could fix would also have been lost.

The sulphuric anhydride was estimated in 100 c.c. of a water containing sulphates of calcium and magnesium; in the residue of the same quantity of water after ignition; and in the ash obtained from 100 c.c., to which 4 grms. of cane-sugar were added:

	Grm.
SO_3 in water direct	0·076
,, residue ignited	0·076
,, ash	0·063

In the experiments in which organic matter was present, the mass was first charred over a bunsen for as short a time as possible, and then incinerated in a muffle.

The conclusions arrived at are—(1) That although chlorides are decomposed and chlorine is lost on incinerating organic substances containing chlorides, the sulphates of calcium, potassium, and sodium, are not appreciably reduced. (2) Magnesium sulphate undergoes decomposition on ignition unless a carbonate is present. In the absence of a carbonate the ash of substances containing magnesium sulphate will, when ignited, consist of magnesium oxide. (3) The quantity of magnesium sulphate in a substance containing chlorides may be sufficient to cause the total loss of the chlorine on incineration.

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NOTE ON THE PRESENCE OF SULPHATES IN FLOUR.

By R. A. CRIPPS, F.I.C., and A. G. WRIGHT.

(*Read at the Meeting, June* 3, 1914.)

During the hearing of a case under the Food and Drugs Acts in Hull last year (Hull Corporation *v.* Thirsk and Sons), in which the defendants were prosecuted for the addition of 6·3 parts of potassium persulphate to 100,000 of flour, the question whether sulphates exist naturally in flour, or whether the sulphur is wholly in organic combination, became somewhat prominent. In consequence of the fact that the presence or absence of sulphates may be of importance in future cases of the kind, we think it may be useful to record the results of some simple experiments. It is common knowledge that the greater proportion of the sulphur in flour is present in organic combination as proteins, and it has been assumed and stated in well-known text-books that the determination of SO_3 in the ash is valueless, because some of the organic sulphur combines with bases and is found in the ash as sulphate. As a quantitative test the determination of SO_3 in the ash is certainly valueless, but because it is too low, not too high. By reference to tables of analyses of the ash of flour one can scarcely fail to notice that the P_2O_5 is greatly in excess of the amount required to form *normal* phosphates with the bases present; hence the ash must be regarded as consisting mainly of *acid* phosphates. Such acid phosphates when heated to a dull redness with the sulphate (or sulphide formed during incineration) readily decompose the sulphate and form phosphate; hence the extremely small proportion of SO_3 usually recorded in the ash of flour (usually below 0·02 per cent. of the ash).

This theoretical opinion was confirmed by gently igniting the mono- and di-acid phosphates of potassium with 1 per cent. of potassium sulphate and with 1 per cent. of calcium sulphate; in each case the resulting salt was found to be free from sulphate.

Having thus indicated the cause of the untrustworthiness of SO_3 determinations in the ash of flour, we made direct estimation of the SO_3 in six samples by the following method: One hundred grms. of flour were shaken with 1,000 c.c. of 1 per cent. acetic acid for one hour, allowed to stand all night, 500 c.c. decanted and boiled with a little HCl, nearly neutralised, and proteins precipitated by Almén's tannin solution, filtered, washed, and the SO_3 determined as $BaSO_4$ in the filtrate after concentration. The following were the results obtained: SO_3 per cent. 0·0069, 0·0076, 0·0084, 0·0069; 0·0084 and 0·0103—the last being whole-meal. Although small, these results are very largely in excess of those recorded in tables based upon the ash, and amount to from 6·9 to 10·3 parts per 100,000.

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THE ESTIMATION OF HYDROCYANIC ACID IN FEEDING-STUFFS, AND ITS OCCURRENCE IN MILLET AND GUINEA CORN.

By J. R. FURLONG, Ph.D.

In estimating the hydrocyanic acid yielded on hydrolysis by various feeding-stuffs, it sometimes happens that, owing to the disturbing influence of the hydrolytic and distillation products accompanying the hydrocyanic acid, it is not possible to employ the iodine or silver nitrate titration methods. The following method, which has been devised to obviate this difficulty, consists essentially of the conversion of the hydrocyanic acid into prussian blue and comparison of the resulting coloration with standards.

One hundred grms. of the ground material is extracted in a Soxhlet apparatus for three hours with 85 to 90 per cent. alcohol. The extract is transferred to a 500 c.c. distillation flask and the alcohol distilled off. By means of a tap funnel 150 c.c. of 10 per cent. sulphuric acid are run in, and a moderately brisk distillation is commenced. The end of the condenser (a Davis' condenser in a vertical position has been found most satisfactory) dips into 5 c.c. of 10 per cent. potassium hydroxide solution. At the end of one hour the receiver is changed, water run into the distillation flask, and the distillation continued. One-hourly fractions are collected until the evolution of hydrocyanic acid ceases.

The distillate is concentrated on the water-bath to 15 c.c., and is then transferred to a boiling tube and treated with 1 c.c. of an iron solution, containing 20 per cent. of ferrous sulphate and 1 per cent. of ferric chloride. The mixture is heated for ten minutes in a boiling water-bath with frequent agitation, and is then allowed to cool, and acidified with concentrated hydrochloric acid. The mixture is again warmed to bring the excess of iron into solution, and, after the addition of 10 c.c. of glycerol, it is allowed to stand until the next day. It is then transferred to a stoppered measuring cylinder and the volume made up to 50 c.c. with water. The mixture is thoroughly shaken, the glycerol forming a colloidal solution with the prussian blue, and the colour is compared in tubes of 1 inch diameter with standards against a white background. It has been found that no hydrocyanic acid is lost during these operations, provided that the amount present is not less than 0·001 grm., but in other cases an appreciable loss takes place in the course of evaporation. The mode of eliminating the possibility of error from this source is explained below.

Six standard tubes are prepared from a solution of potassium cyanide, standardised by means of silver nitrate so as to contain 100 mgrms. of hydrocyanic acid per litre. Portions of this solution, representing 0·002, 0·0015, and 0·001 grm. of hydrocyanic acid, are converted into prussian blue and made up to a volume of 50 c.c. as described above. It is not necessary to distil and evaporate, since, as has been stated already, no hydrocyanic acid is lost during these operations with the quantities mentioned. In preparing standards containing quantities of 0·0008, 0·0005, and 0·0001 grm., however, it is necessary to dilute to 150 c.c. and concentrate as in the preparation of the solution from the plant material.

If the solution to be compared is deeper in colour than the 0·002 grm. standard, it is diluted with 10 per cent. glycerol solution till it matches, the hydrocyanic acid being directly proportional to the volume. If below 0·002 grm. the value is obtained by comparing the solution with the remaining five tubes. It has been found that, whilst the dilution method is applicable to quantities of from 0·01 to 0·002 grm. hydrocyanic acid, it does not hold good for amounts below the latter figure.

In order to obtain consistent results it is necessary to employ a large excess of iron, for whereas 0·25 c.c. of iron solution with 0·002 grm. of hydrocyanic acid gives variable tints, 1 c.c. of iron solution gives concordant colours. The colour due to the excess of iron is minimised on diluting, and if necessary it may be compensated by placing behind the standard a tube of ferric chloride solution of requisite intensity, without impairing the correctness of the result. This also applies to yellow distillates, which are frequently obtained when examining foodstuffs.

The standard colours which were made a year ago have not deteriorated, and are not affected by light.

The method is designed for the estimation of hydrocyanic acid in quantities of 0·1 mgrm. and upwards in such materials as beans and fodders, in which products Dunstan, Henry and Auld (*Proc. Roy. Soc.*, 1903, **72**, 285; 1906 (Ser. B), **78**, 145, 152; 1907 (Ser. B), **79**, 315) have shown that the hydrocyanic acid is present in the form of cyanogenetic glucosides, easily soluble in 85 per cent. alcohol. To provide for the occurrence of free hydrocyanic acid or a cyanogenetic body insoluble in alcohol, the material should be also subjected to steam distillation in the presence of sulphuric acid, but the hydrocyanic acid so obtained is generally less than that yielded by the alcoholic extraction method.

Millet and guinea corn plants from Northern Nigeria examined recently at the Imperial Institute by the above method gave the following results:

Plant.	Stage of Growth.	Percentage of HCN.
uinea corn ...	6 inches high	0·004
	12 „	0·01
	18 „	0·009
	24 „	0·0025
	8 feet, full-grown, but unripe	nil
	8 feet, ripe	nil
illet	6 inches high	0·006
	12 „	0·018
	18 „	0·022
	24 „	0·045
	8 feet 6 inches, full-grown, but unripe	nil
	11 feet, full-grown and ripe	nil

It will be seen from the above table that all the young plants contained a cyanogenetic glucoside. In the case of the guinea corn, the glucoside content reached a maximum in the 12-inch plants, and then decreased as growth proceeded. The millet plants contained a greater amount of glucoside than the guinea corn of a corresponding age, the amount increasing with age up to the largest of the young plants examined. The full-grown plants were free from cyanogenetic bodies.

These results afford interesting indications as to the probable course of the development and disappearance of the cyanogenetic glucosides in millet and guinea corn plants, but it does not appear safe to draw any general conclusions until a larger number of samples has been investigated.

* * * * *

THE DETECTION OF ADDED WATER IN MILK.

By J. WALTER LEATHER, V.D., Ph.D., F.I.C.

Although attention has been drawn by a number of Continental and other chemists (J. Cornalba, *Chem. Zeit.*, 1907-1909; Ducross and Imbert, *Bull. Sci. Pharm.*, 1905; Beckmann and Iordis, *Forsch. ver. w. Lebenson*, 1895, vol. ii.; Barthel, "Methods used in the Examination of Milk and Dairy Produce," 1910; Henderson, *Austral. Assoc. for the Adv. of Sci.*, 1909) to the uniformity in the freezing-point of milk and the value of this test for the detection of added water, there does not appear to have been any communication on the subject to this Society.

Recently I had an opportunity of determining the freezing-point of a number of cow's and buffalo's milks at Lyallpur and at Peshawar, as well as at this Institute, and there seems no doubt as to the great reliability of the test.

Among twenty-five individual cows the freezing-points of the milks varied from −0·529° to −0·577° C.; among the milks of nine buffaloes the variation was from −0·532° to −0·564° C. The freezing-points of the mixed milks of the herds were as follows:

Pusa cow's milk	−0·555° C.
Lyallpur cow's milk	−0·547° C.
Peshawar cow's milk...	−0·537° C.
Peshawar buffalo's milk	−0·552° C.

A freezing-point of −0·537° C. (Peshawar) would indicate, according to Winter's table, 2·36 per cent. added water.

Water was added to Pusa cows' milk in known amounts, and the following are the data obtained:

	Added Water from Winter's Table.	Actually Added.
(*a*) −0·519° C.	5·6 per cent.	5·0 per cent.
(*b*) −0·490° C.	10·9 ,,	10·0 ,,
(*c*) −0·445° C.	19·0 ,,	20·0 ,,
(*d*) −0·433° C.	21·3 ,,	25·0 ,,

Thus in my experience this method is more reliable for the purpose named than any other.

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Estimation of Acetanilide and Phenacetin in Admixture, W. O. Emery. (*J. Ind. and Eng. Chem.*, 1914, **6**, 665-669.)—The method proposed depends on the fact that, when phenacetin is added to a solution of iodine in potassium iodide containing a mineral acid, an iodine addition product or periodide separates out in a crystalline condition, whilst the corresponding product of acetanilide, if formed at all, remains soluble. A quantity of 0·2 grm. of the phenacetin-acetanilide mixture is dissolved by warming with 2 c.c. of glacial acetic acid and 40 c.c. of water, and the solution is poured into a 100 c.c. flask containing 25 c.c. of $\frac{N}{5}$ iodine solution warmed previously to 40° C. Three c.c. of concentrated hydrochloric acid are then added, the closed flask is shaken gently until a crystalline precipitate appears, and the mixture is allowed to cool. After dilution with water to about 97 c.c., the mixture is placed aside overnight, then diluted to 100 c.c., filtered, and 50 c.c. of the filtrate are titrated with thiosulphate solution. Each c.c. of $\frac{N}{10}$ iodine solution is equivalent to 0·00889 grm. of phenacetin. The latter may also be estimated gravimetrically by collecting all the iodine precipitate on the filter, washing it with iodine solution, transferring it to a separating funnel and extracting the phenacetin with chloroform after the free and combined iodine have been destroyed by the addition of sodium sulphite. The acetanilide is estimated in the filtrate from the iodine precipitate; an aliquot portion of the filtrate is treated with sodium sulphite, a slight excess of sodium bicarbonate is added, then 2 drops of acetic anhydride, and the solution is extracted with chloroform. The chloroform extract is filtered, evaporated to 20 c.c., 10 c.c. of dilute sulphuric acid are added, the mixture is heated on a water-bath until reduced to one-half its volume, and, after a further heating for one hour with 20 c.c. of water and 10 c.c. of concentrated hydrochloric acid, the solution is titrated with potassium bromide-bromate solution; each c.c. of the latter should be equivalent to 0·005–0·01 grm. of acetanilide.

When the phenacetin and acetanilide are mixed with caffeine and antipyrine, the mixture of the four substances is digested with dilute sulphuric acid, in order to convert the phenacetin and acetanilide into phenetidine and aniline sulphates, respectively, from which caffeine and antipyrine may be separated by means of chloroform. The phenacetin and acetanilide are then regenerated by treating the acid aqueous solution of the sulphates with sodium bicarbonate in slight excess, adding a few drops of acetic anhydride, and extracting the solution with chloroform.

W. P. S.

Detection of Methyl Alcohol in Spirits. R. Vivario. (*J. Pharm. Chim.*, 1914, **10**, 145-147.)—The spirit is treated with anhydrous sodium carbonate and distilled, so as to obtain a distillate containing about 80 per cent. of alcohol. About

30 grms. of this distillate are boiled for seven hours beneath a reflux condenser with about 15 grms. of potassium hydroxide and 1 grm. of hydroxylamine hydrochloride. The liquid is then cooled, rendered slightly acid with sulphuric acid, and distilled in a current of steam. In the presence of methyl alcohol or its derivatives, the distillate will contain hydrocyanic acid, which may be identified by the Prussian blue reaction, whereas no hydrocyanic acid is formed under these conditions by alcohol, acetaldehyde, acetone, acetal, or amyl alcohol. The test is capable of detecting less than 10 per cent. of methyl alcohol in spirits of wine. Furfural, if present in any considerable quantity, should be eliminated by treatment with metaphenylene-diamine hydrochloride, or aniline phosphate. Spirits, such as gin, brandy, and rum, should be distilled, and the test applied to the first 30 c.c. of the distillate. C. A. M.

Analysis of Stabilised Cacao. L. Reutter. (*Schweiz. Apoth. Zeit.*, 1914, **52**, 308-312; 322-325; 335-339; through *Chem. Zentralbl.*, 1914, II., 169-170.)—By stabilisation is meant the prevention of fermentation in freshly gathered cacao beans. The pods were formerly allowed to undergo fermentation in order that the husk might be readily removed, but this fermentation has a considerable effect on the beans themselves. The pods may, however, be treated with a 1 per cent. soda solution at 45° C.; the husk is then easily removed, and the beans may be sterilised. The cacao thus obtained does not contain any preformed sugars, these, together with theobromine and tannin, being products of hydrolysis. Theobromine does not occur in the free state in cacao, caffeine is absent, and only traces of ethereal oils are present. From the dilute methyl alcohol extract of defatted cacao there may be obtained a red-violet substance—cacaorin—having the formula $C_{10}H_{20}O_6N_8$, and cacao red, of which the formula is $C_{40}H_{60}NO_{27}$. On hydrolysis, cacao red yields a dextro rotatory sugar and an insoluble substance, cacao brown, having the formula $C_{76}H_{78}NO_{34}$. W. P. S.

Cacao Butter Substitute. C. Grimme. (*Gordian*, 1914, **19**, 6971-6973.)—An artificial fat mixture, giving on analysis practically the same analytical values as pure cacao butter, appears to have been extensively offered for sale on the Continent in place of the genuine article. The specific gravity, melting-point, refractive index, saponification value and iodine value of the artificial fat are the same as those of typical specimens of cacao butter, and there is sufficient cacao butter in the spurious article to impart to it the characteristic smell of the genuine product. Certain differences, however, in the crystalline appearance of the fatty acids and in their solidifying-points, and also in the critical temperatures of solution of the fats in alcohol and acetic acid, may be made use of in the detection of the artificial mixture. The values obtained were as follows:

		Genuine Cacao Butter.	Artificial Fat Mixture.
Solidifying-point of fatty acids	... ...	48·8°–49·2° C.	44·9°–45° C.
Critical temperature of solution	In 2 vols. absolute alcohol ...	79·5°–80·5° C.	76° C.
	In 1 vol. glacial acetic acid ...	65°–69° C.	35° C.

Fractional crystallisation of the glycerides from ether-alcohol solution and determination of the melting-points of the fractions obtained is also of value in confirming the presence of foreign glycerides in a sample of cacao butter. Pure cacao butter, after two recrystallisations from ether-alcohol, gave crystals melting at 56° to 57·4° C., while the crystals obtained in exactly the same way from the artificial mixture melted at 39·5° to 40° C. G. W. M.-W.

Comparative Study of Different Methods of Determining Cane-Sugar in Beet Products by the Double Polarisation Process. R. Gillet. (*Bull. Assoc. Chim. Sucr. Dist.*, 1914, **31**, 992-1033.)—If in the double polarisation method the direct reading be taken in an alkaline solution (due to the basic lead acetate used for clarification) and the inversion reading in an acid medium (due to the hydrochloric acid used for inversion), an error is introduced owing to the change of rotation of the optically active amino-acids (ANALYST, 1911, **36**, 120). When the direct reading is made with a solution containing the same amount of hydrochloric acid as that used for inversion in the manner recommended by Andrlik (*Zeitsch. Zuckerind. Böhm.*, 1906, 417), urea being added to retard hydrolysis while making the observation, exact results are obtained, but the process is difficult to operate. Equally reliable, but more practical, is the procedure first recommended by Pellet of making the direct reading in the presence of an excess of sulphurous acid (ANALYST, 1913, **38**, 282). Making the direct and inversion readings in a neutral medium according to Saillard's process (*Bull. Syndic. Fabr. Sucre.*, 1913, No. 62) is not practical. With the invertase process (ANALYST, 1911, **36**, 120; 1913, **38**, 281) results closely agreeing with the Andrlik and Pellet modifications are obtained, though it is too tedious for routine work. In the author's opinion the most exact and the simplest method of operating double polarisation is that in which the direct reading is made in a solution containing sulphurous acid in excess, and for general work it only should be used. J. P. O.

Correlation between the Specific Characters of the Tasmanian and Australian Eucalypts. R. T. Baker and H. G. Smith. (*British Association Report*, 1914, No. 137.)—The authors review the results of their recent research on Tasmanian eucalypts, comparing them with their earlier work on the eucalypts of the mainland, supplemented by more recent data. The ground covered by these investigations, now extending over a period of a quarter of a century, embraces almost the whole geographical range of the genus—an area of the earth's surface of about 3,000,000 square miles. Such an area includes a diversity of soils, climates, altitudes, etc., and naturally there are a great variety of species; but it is found at the same time that a relative constancy of specific botanical features and chemical constituents characterises the whole genus.

Comparisons as well as contrasts are made of the morphological and chemical features of the trees found at the sea-level and right up to the highest altitudes at which the correlated species occur both in Australia and Tasmania.

Study of Authentic Samples of Gum Turpentine. A. W. Schorger. (*J. Ind. and Eng. Chem.*, 1914, **6**, 541-548.)—In recent years "wood turpentine," obtained by steam or destructive distillation of resinous wood, has been placed on the market. In consequence, the United States Department of Agriculture considered it necessary to obtain additional and unimpeachable data concerning the physical properties of genuine turpentine derived from collected oleo-resin. Accordingly a number of samples were collected at distilleries from Mississippi to Georgia by officials of the Department, who saw the raw materials collected and the distillation carried out. Most of the gum was derived from *Pinus palustris* or *P. heterophylla*. There is a tendency for the sp. gr. of the distillate from "scrape" to be higher than that from "dip," higher for turpentines collected in the spring than for those collected in the autumn, higher for old faces than for new, higher for gum collected in "boxes" than for gum collected in "cups," and naturally higher for the final fractions than for the first. However, the differences between the distillates from all these sources are slight. The properties of bulk quantities should approach the values given below. Turpentine stored with reasonable care for a year shows but little change. Normal, freshly distilled (American) turpentine has a sp. gr. between 0·866 and 0·872 (average, 0·869), an index of refraction between 1·471 and 1·475 (average, 1·473), and begins to boil at 154° to 157·6° C. (average, 156° C.). The first fraction of 5 per cent. has a sp. gr. not below 0·864 (average, 0·865), and a refractive index not below 1·469 (average, 1·470). At least 86 per cent., generally 91 to 93 per cent., distils below 165° C.; and at least 93 per cent., generally 97 per cent., distils below 170° C. If distillation be stopped when 5 per cent. remains in the still, the residue will have a sp. gr. not below 0·887 (average, 0·914). In no case should the sp. gr. of any fraction be lower than that of the previous fraction. G. C. J.

Estimation of Esters in Essential Oils. J. Nivière. (*Bull. Soc. Chim.*, 1914, **15**, 677-680.)—The saponification values of various essential oils and esters estimated in the usual way in a flask with a long condensing tube agreed with those obtained beneath a reflux water condenser. The results obtained by saponifying bergamot and lavender oils under pressure, as suggested by Helbing, were 2 to 3 units higher than those given by the open-tube method; but estimations with linalol and linalyl acetate showed that these did not cause the difference. On the other hand, the presence of an aldehyde raised the saponification value in the closed flask method. Hence the ordinary method gives a more accurate estimation of the amount of esters. C. A. M.

New German Standards for Malt Analysis. G. Bode and A. Wlokka. (*Allgem. Zeitsch. Bierbrau. und Malzfab.*, 1914, **42**, 291-295.)—The Verein Deutscher Chemiker has confirmed the recommendations of its Malt Analysis Sub-Committee, which will take effect from September 1, 1914, and be known as the "Bonn Standards."

Samples may be taken either with a borer—*e.g.*, Barth-Eckhardt's, with separate closing compartments—or with an automatic sampler, such as those of Gräfe and Wlokka. If the malt is in sacks, 10 per cent. of the sacks are to be sampled; if in bulk, the heaps are to be shovelled over, and samples drawn at different depths. The samples must be mixed in a dry receptacle, and then packed in well-closed bottles or tins. Not less than 400 grms. are to be sent to the analyst, who may remove large foreign bodies (stones, wood, string), but not dust nor foreign seeds.

Grading.—For grading grist or meal, a machine containing five sieves, constructed by Seck to the specification of the Pfungstadt brewery, must be employed. A complete description of the sieves is given. The mean linear dimensions of the meshes are—1·27, 1·01, 0·547; 0·253, and 0·152 mm. One hundred grms. of grist or 50 grms. of meal are shaken on the sieves for five minutes at a speed of 300 revolutions per minute. Small rubber balls are placed in the three lowest sieves.

Grinding.—Mills constructed by Seck are specified. The adjustment of the mills is to be standardised by means of the sifting apparatus, and a standard well-modified malt (0·2 to 0·25 on Brand's colour scale), containing 4 per cent. of moisture, and showing a difference of from 0·6 to 1·0 per cent. between the extract yields from grist and meal. For estimations of extract, the setting of the grist mill is correct when the last three sifted fractions total 40±1 per cent.; this corresponds to a clearance of about 1 mm. between the rolls. The fine mill is to be adjusted so as to yield 90±1 per cent. in the three finest fractions. Grist for the estimation of moisture must be ground in a special hand-mill, and yield 60 per cent. in the last three fractions. For trade analyses the moisture and extract are to be determined only in the meal.

Moisture.—The meal mill is first swept out by passing through it 5 grms. of the malt to be analysed; then about 10 grms. are ground to meal, and divided without loss into two portions of 5 grms. each, which are weighed accurately into drying tubes 40 mm. in diameter and 20 to 30 mm. high. The time of drying in the oven, previously adjusted above 100° C., is three hours. The drying oven must be of approved type; designs by Scholvien, Ulsch, and V.L.B. have been sanctioned. The efficiency of the oven must be tested once a year with four types of malt of very different constitution. The temperature must be above 100° C., and may be first tried at 103° to 104° C. The difference between duplicate estimations must not exceed 0·1 per cent., and a further period of heating for one hour must not produce a further loss exceeding 0·1 per cent. Should these conditions not be fulfilled, the tests should be repeated at a temperature 1° C. higher, but in no case above 108° C.

Extract.—Mashing must be performed with mechanical stirring. The stirrer blades must not touch the sides of the beaker, and must clear the bottom by 1 to 2 mm. The beakers and stirrers must be of the same metal, not nickel-plated. All the stirrers must work at the same speed—80 to 100 revolutions per minute. For the estimation, 50 to 51 grms. of malt are ground directly into the beaker, the mill is brushed out, and the quantity of meal adjusted to exactly 50 grms.; 200 c.c. of distilled water at 45° to 46° C. are stirred in by hand, and the beaker is then placed in the mashing-bath, previously heated to 45° C. The mash is stirred at this temperature for thirty minutes, and the bath then heated up, at the rate of 1° C. per minute, to 70° C.; 100 c.c. of water at 70° C. are added, and mashing is continued for one hour. The whole is cooled within ten to fifteen minutes to the ordinary temperature, and the mash made up with water to 450 grms. The whole mash is poured on a C.S. and S. folded filter, No. 560, of 32 cm. diameter; the first 100 c.c. of filtrate are returned. Filtration is not to be continued for more than two hours. The sp. gr. of the wort at 17·5° C. is determined in a pyknometer of the following dimensions: Capacity, about 50 c.c.; total height, 140 to 160 mm.; length of neck, 65 to 85 mm.; internal width of neck, 2·5 to 4·0 mm.; mark, 25 to 35 mm. from top edge. The filled pyknometer is immersed up to the mark in a water-bath at 17·5° C., and allowed to return to the temperature of the room before weighing. Results from duplicate beakers must agree within 0·0002. The values are calculated by Balling's table.

Saccharification.—The time of saccharification is observed by placing a drop from the mash on a gypsum plate ten minutes after reaching 70° C., and testing with $\frac{N}{50}$ iodine. The spot-test is repeated every five minutes until the colour is pure yellow. If saccharification is incomplete after one hour, a separate mashing is made at 75° C. for this test only.

Colour.—The colour of the wort is expressed in terms of Brand's dyestuff solution, based on $\frac{N}{10}$ iodine, to be obtained fresh every year from the Munich station.

Estimation of extract from grist, also the mechanical analysis, comprising hectolitre weight, 1,000 corn weight, and modification of endosperm, are only to be performed by special request. The measurement of the acrospire is abandoned.

Results from different laboratories must agree within 0·25 per cent. for moisture, and within 0·6 per cent. (including the error for moisture) for extract in dry substance.

J. F. B.

Detection of Morphine and Phenols by Means of Uranium Salts. J. Aloy and C. Rabaut. (*Bull. Soc. Chim.*, 1914, **15**, 680-682.)—Solutions of uranium salts form a red compound with compounds containing a free phenolic group, and in this way morphine may be distinguished from the other opium alkaloids and from bases not containing a free phenolic group. Thus the reaction is given by guaiacol and official sodium salicylate, but not by anisol, veratrol, phenetol, heroine, and neutral sodium salicylate. In testing for morphine, a small quantity of the alkaloid is shaken with a few drops of methyl alcohol and a crystal of uranium nitrate; or morphine hydrochloride is dissolved in water, and the solution shaken with a few drops of a saturated solution of uranium acetate. A red coloration is obtained, which disappears on the addition of an acid, while on evaporating the solution a red residue is left. The reaction will detect 0·0001 grm. of morphine. Aqueous solutions of di- and tri-phenols give a red coloration, which is more intense in proportion to the number of hydroxyl groups. The presence of chlorine and bromine in the compound does not interfere with the reaction, which is prevented, however, by sulpho and nitro groups.

C. A. M.

Essential Oil of *Ocymum Pilosum, Roxb.* K. Bhaduri. (*J. Amer. Chem. Soc.*, 1914, **36**, 1772-1773.)—The seeds of *Ocymum pilosum*, Roxb., when steeped in water, swell into a jelly, which is used medicinally by the natives of India. The plant has a strong odour of the essential oil, which is found in the whole of it, including the seeds. When the latter are dried, no oil can be obtained from them, but green seeds contain more oil than any other portion of the plant. The following analytical figures relate to a single sample of the oil obtained by submitting the whole plant to distillation in a current of steam. The oil, which resembled lemon-grass oil in odour, had a sp. gr. of 0·8872 at 25·5° C., a refractive index of 1·4843 at 24·5° C., and $[\alpha]_D$ 4·17° at 24·5° C. It contained approximately 41 per cent. of citral, 34 per cent. of citronellal, together with smaller quantities of cineol and limonene, and a very small amount of thymol. It does not contain methyl-chavicol, which is generally present in Basil oils. G. C. J.

Pharmaceutical Chemical Standards. C. A. Hill. (*Chemist and Druggist*, 1914, **85**, 49-55.)—In view of the fact that definite standards of purity in respect of chemicals have been officially recommended by the Committee of Reference in Pharmacy to the General Medical Council for inclusion in the next edition of the British Pharmacopœia, the author records the results of the examination of a large number of samples of pharmaceutical chemicals obtained during the past four years. These results are given in tabular form, and mention is made of the number of samples which have been found to conform with a specified limit of purity, together with the highest and lowest amounts of any impurity present. The author considers that 20 parts per million would be a suitable limit for the amount of lead admissible in boric acid, but that the limit of 20 parts per million for this metal in tartaric acid, citric acid, and cream of tartar, is rather too stringent. W. P. S.

Estimation of Podophyllum Resin. W. M. Jenkins. (*J. Ind. and Eng. Chem.*, 1914, **6**, 671-672.)—Podophyllum resin is slightly soluble in water, and this solubility is greatly increased by the presence of small quantities of alcohol; therefore methods which depend on the precipitation of the resin by diluting its alcoholic solution with water yield inaccurate results. The resin is, however, readily soluble in a mixture of 1 part of alcohol and 2 parts of chloroform, although only partially soluble in the latter solvent alone, and the following method, based on these facts, is proposed for the estimation of the resin in its liquid preparations; 99 per cent. of the quantity of resin present is recovered. Five c.c. of the sample are shaken in a separating funnel with 5 c.c. of alcohol, 10 c.c. of chloroform, and 10 c.c. of water containing 0·6 per cent. of hydrochloric acid (2 c.c. of concentrated acid per 100 c.c. of water); when separation has taken place, the lower layer is drawn off and the contents of the funnel are extracted twice more with the mixture of alcohol and chloroform, using 15 c.c. each time. The combined extractions are shaken with 10 c.c. of the dilute hydrochloric acid, the lower layer is drawn off into a weighed flask, and the acid wash-water is extracted twice with alcohol-chloroform. The solution in the flask is then evaporated, the residue dried at 100° C., and weighed.

For the analysis of the drug itself, 10 grms. of the powder are boiled for three hours under a reflux apparatus with 25 c.c. of alcohol; the mixture is then transferred to a small percolator and washed with alcohol until about 50 c.c. of percolate are obtained. This solution is cooled, diluted with alcohol to 50 c.c., and 10 c.c. of it are employed for the estimation of the resin. W. P. S.

Detection of Saponin in Aerated Waters. N. Campos. (*Ann. Chim. anal. applic.*, 1914, **19**, 289-293.)—The method of Vamvakas (ANALYST, 1906, **31**, 232) is not reliable in the case of many kinds of mineral waters, and has the drawback that several of the reactions described as distinctive of saponin are also given by an extract of coffee. The following modification of the method of Rühle is more generally applicable: A definite quantity (not less than 500 c.c.) of the sample is evaporated to 10 c.c. on the water-bath, and then shaken in a stoppered tube with 10 c.c. of melted phenol and 20 grms. of ammonium sulphate. The phenolic layer (containing the saponin) is separated, and shaken with 100 c.c. of ether and 50 c.c. of water, and the aqueous extract of the saponin is separated and concentrated. The residue of saponin is still too impure to give the sulphuric acid reaction, but may be separated in the form of a stable compound with barium, which is soluble in water but insoluble in concentrated solutions of barium hydroxide. For this purpose the concentrated aqueous extract (25 c.c.) is shaken with 100 c.c. of a freshly saturated solution of barium hydroxide, and the clear liquid decanted from the deposit, and shaken with a fresh portion of barium hydroxide solution. This treatment is repeated several times until the liquid is completely decolorised. Finally, the united precipitates are decomposed by means of a slow current of carbon dioxide, and the liquid filtered. The filtrate, containing the saponin, is evaporated on the water-bath, and the residue (which will contain some barium carbonate) is extracted with several successive portions of boiling alcohol. These extracts are allowed to evaporate, and leave residues of increasing purity, to the last of which the colour tests for saponin may be applied. In the case of other residues the colorations with sulphuric acid will range from green to yellow, rose, and reddish-violet. C. A. M.

Unsaponifiable Constituents of Shea Butter and their Relation to those of Mowrah Fat. P. Berg and J. Angerhausen. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1914, **28**, 73-79.)—The authors have shown recently (ANALYST, 1914, 310) that mowrah fat contains unsaponifiable substances having a specific rotation of about $[\alpha]_D+35°$. Examination of shea butter shows that this fat also contains optically active substances. The total unsaponifiable substances amount to about 6 per cent., of which more than one-half is soluble in alcohol, and is not precipitated by digitonin. This portion has a specific rotation of about $[\alpha]_D+38°$. In this respect shea butter is similar to mowrah fat. W. P. S.

Fractional Liquefaction of Rice Starch. F. J. Warth and D. B. Darabsett. (*Mem. Dept. Agric. in India.*, *Chemical Series*, 1914, **3**, 135-146.)—In a comparative investigation of different varieties of rice, the authors studied the fractional liquefaction of the starches by hot water at different temperatures. A definite weight (usually 1 grm.) of the finely divided material was stirred with 70 c.c. of water, and maintained at the desired temperature for one hour. All the starch susceptible to liquefaction at that temperature was thus converted into a paste containing the unaltered granules in suspension. The liquid was cooled, water was added to compensate for evaporation, and the paste then digested with 10 c.c. of malt extract at 30° C. for two hours. The solution was made up to a definite volume, filtered with kaolin, and an aliquot portion of the filtrate hydrolysed with hydrochloric acid. The dextrose was estimated by Fehling's solution, the cuprous oxide being determined by the permanganate method. On the assumption that the ungelatinised starch resisted the action of the diastase, the amount of starch liquefied at a given temperature was calculated from the dextrose value. In the preliminary disintegration of the rice, mechanical grinding was found to increase the liquefaction of the starch to an extent depending on the bruising of the granules, but constant results were obtained by steeping the weighed polished rice in 10 c.c. of 1 per cent. potassium hydroxide for twenty-four hours, then acidifying slightly and pouring off the liquor into a beaker. The steeped grain was then gently crushed in a mortar with successive quantities of water, the starchy liquor being collected with the rest. The acidity was neutralised with suspended calcium carbonate, and liquefaction carried out as described above. From a comparison of the results from seven varieties of rice, it was found that the nature of the starch varied with the cooking quality of the grain. Three specimens of hard vitreous varieties showed great similarity. Up to 65° C., liquefaction was very slight; at 70° C., when other rice starches were almost completely liquefied, these showed only about 20 per cent.; then between 70° and 75° C. there was a sudden breakdown of all but a small residue of resistant starch. The opaque white glutinous rices contained a considerable proportion of starch liquefied at 65° C., and practically no resistant starch above 75° C. Intermediate varieties showed varying proportions of the 60° to 65° C., the 70° to 75° C., and the more resistant modifications. J. F. B.

Proximate Analysis of Wheat. N. A. Barbieri. (*Comptes rend.*, 1914, **159**, 431-434.)—Twenty kilos of wheat (*T. turgidum*) were submitted to a process of fractional analysis as follows: (1) Washing with cold distilled water. The wash-waters, which were very slightly coloured, were concentrated on the water-bath and shaken with chloroform, the two layers being separated. (2) Steeping for fifteen hours in chloroform-water. The concentrated extract, which was strongly coloured, was also shaken with chloroform. (3) Extraction with water at 50° to 60° C., without breaking the cortex. The concentrated extract was precipitated with alcohol and filtered. (4) Extraction with boiling water for one hour. The grains swelled and burst; the liquid was drained off and precipitated by alcohol. (5) The burst grains were treated on a sieve, and the portion which passed through was dehydrated with strong alcohol, thus forming a meal free from bran. (6) The bran and germs left on the sieve were also dehydrated with alcohol. (7) The aqueous-alcoholic liquors from 3, 4, 5, and 6, were united, concentrated, filtered, and shaken with chloroform. The chloroform extract, mixed with those from 1 and 2, was evaporated, the residue taken up with ether, the ethereal solution filtered and evaporated. The residue was taken up with absolute alcohol, and the filtered alcoholic extract evaporated *in vacuo.* There were thus obtained 2 grms. of bléine, the yellow colouring matter of the wheat-corn, probably of chlorophyllic origin. Bléine is soluble in benzene, carbon disulphide, light petroleum, etc. It shows neither the biuret nor the acrolein reaction. The aqueous portion of this fraction was treated with alcohol until a slight cloudiness was formed, and then submitted to dialysis. The dialysed liquid, when treated with excess of acetone, gave a precipitate of salts consisting of potassium dihydrogen phosphate and potassium sulphate in about equal quantities. The same salts were obtained also from the aqueous extracts from 1 and 2. (8) The meal from 5 and the precipitates from 3 and 4, on extraction with ether, gave 225 grms. of oil, of which only a small proportion was soluble in acetone. This latter portion contained traces of phosphates, while the portion insoluble in acetone was free from phosphorus. Hence it would appear that the wheat-corn is devoid of lecithins, since these bodies are generally regarded as being insoluble in acetone. (9 and 10) One kilo each of the meal and the bran and germ fractions were incinerated separately. The meal yielded 0·30 per cent. of insoluble salts, and the bran and germs 0·62 per cent. These salts were mainly composed of calcium sulphate and tricalcium phosphate; carbonates were absent. J. F. B.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Estimation of Arsenic in Organic Substances. M. Vinograd. (*J. Amer. Chem. Soc.*, 1914, **36**, 1548-1551.)—In the estimation of arsenic in such substances as blood, spinal fluid, tissue, etc., the preliminary destruction of the organic matter is best attained by heating the material with nitric acid in a sealed tube. Three c.c. of blood, the residue obtained on the evaporation of 100 c.c. of spinal fluid, or 0·5 grm. of dry powdered tissue, are placed in a tube with 1 c.c. of nitric acid, the tube is sealed and heated to 260° C. for an hour or two; the tube is then opened, its contents are rinsed with water into a flask, the solution is evaporated to 15 c.c., 5 c.c. of sulphuric acid are added, and the mixture heated until all the nitric acid has been expelled. The arsenic in the solution is then estimated by the modification of the Gutzeit test described by Sanger and Black (ANALYST, 1908, **33**, 28). W. P. S.

Cyanogenetic Plants of New South Wales. J. M. Petrie. (*British Association Report*, 1914, No. 140.)—Of the plants growing in New South Wales, over a thousand species have been examined for hydrocyanic acid and cyanogenetic glucosides. Sixty of these gave positive results with sodium picrate paper. These include forty-four species native to New South Wales in seventeen natural orders. Some plants, well known to be cyanophoric in Europe, when grown in this State have never given any reaction, although tested in all seasons.

Only a few were found to evolve free hydrocyanic acid naturally, but all showed the presence of a glucoside and enzyme. When the natural enzymes in these plants were killed by boiling water, the reaction to sodium picrate paper ceased; if then a few drops of emulsin, prepared from sweet almonds, were added, positive reactions were again obtained, showing that in all cases the glucosides present in the plants were capable of being hydrolysed by emulsin.

Of the sixty species stated, twenty are grasses, and these include eleven species indigenous to this State. The *Sorghum vulgare*, examined by Dunstan and Henry, was found to lose its glucoside when 14 inches high, while the Australian-grown plant retains it when 4 feet high, and mature. Both glucoside and enzyme slowly disappear with air-drying. One hundred and fifty species of grasses were tested systematically for seasonal variations, and some were found to give negative results at particular seasons. Two species of grasses alone evolved free hydrocyanic acid, and only one of these is available for grazing. This is the only one, except the sorghums, which has been associated with fatalities among stock.

Among the non-cyanogenetic grasses, thirty-three species contained emulsin-like enzymes.

Results of the Hypochlorite Disinfection of Water Supplies. A Comparison of the Reduction of the Different Members of the *Bacillus Coli* Group. J. B. Thomas and E. A. Sandman. (*J. Ind. and Eng. Chem.*, 1914, **6**, 637-639.)—The two supplies were treated with hypochlorite at the impounding reservoirs, then passed through pipes, about a mile in length, to the storage reservoirs, where the water was kept for five to seven days before entering the distribution system. The amounts of available chlorine applied varied from 0·65 to 2 parts per million. Lactose bile was used as an initial medium, and Endo's agar for isolating the *B. coli* organisms in pure culture; these organisms were divided into four subgroups by the use of dulcitol in addition to the commonly-used sugars and gelatine. For the purpose of classification those organisms causing gas-production in lactose, dulcitol, sucrose, and dextrose, were considered to be *B. coli communior*; those causing gas-production in lactose, dulcitol, and dextrose, without liquefaction of gelatine, were classed as *B. coli communis*; those causing gas-production in the three sugars, but not in dulcitol, with or without liquefaction of gelatine, as *B. lactis aerogenes*; and those causing gas-production in lactose and dextrose only were called *B. acidi lactici.* This, however, was but a general classification. The number of organisms growing on standard agar medium was also determined. The treatment caused a reduction of from 97 to 99 per cent. of these four subclasses of organisms, and all appear to be equally vulnerable to the action of chlorine. A greater reduction (83 to 99 per cent.) was observed in the organisms growing on agar at 20° C. than in those growing at 37° C. (76 to 85 per cent.). When the amount of available chlorine added exceeded 1·5 parts per million, the water delivered to the city had an odour of chlorine, and the presence of free chlorine could be detected chemically; the addition of smaller quantities led to fewer complaints from consumers, although trouble was still encountered when the water was used for the manufacture of ice. The odour of chlorine was most noticeable in the core ice where it became segregated as the water froze; distillation of the water before freezing did not eliminate the odour. W. P. S.

Method of Preparing Strong Invertase Solutions from Top and Bottom Yeasts. C. S. Hudson. (*J. Amer. Chem. Soc.*, 1914, **36**, 1566-1571.)—Ten kilos. of the yeast are mixed with 10 litres of tap water and 500 c.c. of toluene, and allowed to stand for from three to five days. Neutral lead acetate is then added until no further precipitate forms, the mixture is filtered, excess of lead is removed from the filtrate by treatment with hydrogen sulphide, and the solution is submitted to dialysis in a collodion sack for about twenty-four hours. The solution may be concentrated under reduced pressure at 39° C., or even evaporated to dryness without decreasing in activity. Bottom fermentation or brewer's yeast yields an invertase solution which is almost twice as strong as that yielded by top fermentation yeast.

W. P. S.

Distribution of Nitrogen in Vegetable, Yeast, and Meat Extracts. F. C. Cook. (*J. Amer. Chem. Soc.*, 1914, **36**, 1551-1556.)—In distinguishing between these three classes of extracts, the quantities of creatinine, ammonia, and purine nitrogen, afford useful data. Plant extracts—that is, extracts which are prepared from soya beans, vegetables, etc., do not contain creatinine or creatine, and only very small quantities of purine bases (from 0·23 to 0·46 per cent. of purine-nitrogen). Creatine is also absent from yeast extracts, but large quantities of purine-nitrogen are present—namely, 11·2 per cent. Meat extracts contain from 3 to 5 per cent. of purine-nitrogen, and from 22 to 32 per cent. of creatinine-nitrogen. All the above results are given as percentages of the total nitrogen. The total nitrogen present in the extracts examined was—Meat extracts, 7·49 to 9·65; yeast extract, 5·35; plant extracts, 5·23 to 6·47—per cent. All the nitrogen of the plant extracts is found in the filtrate from the acid-alcohol precipitate (1 grm. of the extract dissolved in 10 c.c. of water and treated with 1·2 c.c. of 12 per cent. hydrochloric acid and 200 c.c. of alcohol), whilst about 25 per cent. of the nitrogen of meat and yeast extracts is rendered insoluble by this treatment. Of the total nitrogen in plant extracts, from 6 to 13 per cent. is present as ammonia; in meat and yeast extracts the amount varies from 1·5 to 2·6 per cent.

W. P. S.

Commercial Papain and its Analysis. H. M. Adams. (*J. Ind. and Eng. Chem.*, 1914, **6**, 669-670.)—The most frequent adulterants of papain are starch and pepsin. The presence of the former is readily detected by the iodine reaction, whilst digestion experiments are required to detect pepsin. When tested in an acid solution, a small amount of pepsin greatly increases the strength of the papain; but if a neutral or slightly alkaline solution is used, the action of the pepsin is inhibited and the true digestive action of the papain is obtained. For the determination of the activity of papain, raw meat appears to be the best material to be digested, owing to the small residue left after digestion, and a neutral medium gives the most satisfactory results. The following procedure is, therefore, recommended in preference to the method described by Rippetoe (ANALYST, 1912, **37**, 405): Ten grms. of fresh, lean, raw meat, which has been passed through a mincing machine, and 0·325 grm. of the papain, are placed in a 6-ounce bottle, 100 c.c. of water are added, and the mixture is shaken until the meat is disintegrated. The bottle is then placed for five hours in water at 50° to 55° C., and shaken for one minute every fifteen minutes. The contents of the bottle are now poured into a graduated tube, allowed to settle for at least one hour, and the volume of the sediment is observed. A standard should be run at the same time, using a specimen of papain known to have a high digestive strength.

W. P. S.

ORGANIC ANALYSIS.

Estimation of Carbon Monoxide. J. I. Graham and T. F. Winmill. (*J. Chem. Soc.*, 1914, **105**, 1996-2003.)—The method described is a modification of the iodine pentoxide method. As usually carried out, this method requires a large volume of gas, and is inconvenient in other respects. The authors use a modified Haldane's apparatus, with three of the usual pipettes and a U-tube of such dimensions that, when charged with 2 grms. of iodine pentoxide, there is a minimum amount of dead space. One of the ordinary pipettes is charged with bromine dissolved in an aqueous solution of potassium bromide, as unsaturated hydrocarbons must be removed before proceeding to the estimation of carbon monoxide. The other pipettes are charged with strong potash, to absorb any carbon dioxide present in the original gas, as well as the carbon dioxide and iodine vapour resulting from the reaction between carbon monoxide and iodine pentoxide. These pipettes also serve for passing the gas back and forth through the iodine pentoxide tube. This is done four times, the tube being heated to 90° to 100° C. in a beaker of hot water. At lower temperatures the reaction is slow, whilst at higher ones it is more difficult to avoid oxidation of hydrogen. Before and after the oxidation of the carbon monoxide, the U-tube is immersed in cold water, and the volume of gas in the 20-c.c. Haldane burette read, after making the customary adjustments. If the initial and final temperatures are not identical, a correction, readily determined by experiment, is applied; but this correction should not exceed 0·002 c.c. per 1° C. in a well-packed U-tube of suitable dimensions. Hydrogen alone does not appear to react with iodine pentoxide below 130° C., but when carbon monoxide is simultaneously present it is oxidised even at 90° C., the velocity of reaction depending on the concentration of the carbon monoxide and the hydrogen. If the mixture contains less than 20 per cent. of hydrogen and less than 8 per cent. of carbon monoxide, the error involved is less than the unavoidable experimental error of the method—namely, 0·02 per cent. If the concentration of hydrogen is below 1 per cent., as much as 25 per cent. of carbon monoxide can be satisfactorily estimated. Gases rich in hydrogen and carbon monoxide must be diluted with air before analysis. Methane and its homologues do not interfere. G. C. J.

Relation of Composition of Ash in Coal to its Fusing Temperature. J. H. K. Burgwin. (*J. Ind. and Eng. Chem.*, 1914, **6**, 694-695.)—The author agrees with Palmenberg (ANALYST, 1914, 370) that no definite fusing temperature accords with a definite percentage, but does not agree that there is no relation between the clinkering quality of a coal and the sulphur or iron content. On the contrary, he shows that Palmenberg's own figures, when plotted on a system of rectangular co-ordinates, bring out the fact that no ash containing less than 10 per cent. of iron melts below 1399° F., whereas no ash containing more than 20 per cent. has so high a melting-point. Between 10 and 20 per cent., the fusing temperature varies over so wide a range as to justify Palmenberg's dictum with which this abstract opens. His wider generalisation, however, is disproved not only by the above facts, but by plotting the fusing temperatures of the ash against the percentage of sulphur in the coal (not this time in the ash). Without exception, every coal with less than 1 per cent. of sulphur yields an ash melting above 1399° C., whereas every coal with more than 2 per cent. yields an ash melting below this temperature. In a combined diagram, the fusing-point of the ash is plotted against the sum of the percentages of iron and sulphur in the coal. With three exceptions out of forty-five observations, when the percentage of sulphur and iron is less than 3, the fusing-point of the ash is above 1399° C.; and when this percentage exceeds 3, the fusing-point is below 2,550° F., a point below which it may be said in general that it is undesirable and—with even moderately hard driving—impossible to keep the heat of a boiler furnace.

G. C. J.

Fluorescence of Petroleum Distillates. B. T. Brooks and R. F. Bacon. (*J. Ind. and Eng. Chem.*, 1914, **6**, 623-625.)—The fluorescence of petroleum distillates is due to the presence of a substance (or substances) which, for the greater part, is formed during the distillation. By extracting 12 litres of lubricating distillate with strong sulphuric acid, diluting the acid solution, removing the excess of sulphuric acid by the addition of lime, and evaporating the liquid nearly to dryness, a crystalline residue was obtained which, on extraction with alcohol and evaporation of the alcoholic solution, yielded about 1 grm. of crystalline residue. This substance gave an intensely fluorescent solution when dissolved in kerosene, etc., and probably consisted of one or more compounds of the benzene series resembling, or perhaps identical with, chrysene, fluorene, or pyrene. The fluorescence of mineral oils is not due to colloidal suspension of carbon or sulphur. Although oxidising agents destroy the fluorescence, the "deblooming" action of nitro compounds is purely physical in character; when the added nitro compound is removed by treatment with alcohol, the oil again becomes fluorescent. The fluorescent substance is destroyed by halogenation or hydrogenation, but is only slightly absorbed by fuller's earth.

W. P. S.

Method for calculating Viscosity of Mineral Oil Mixtures. E. Molin. (*Chem. Zeit.*, 1914, **38**, 857-859.)—It is shown that Pyhälä's formula (*Petroleum*, 1911, 217) gives widely erroneous results except when the two oils have nearly the same viscosity, or one of them has a value approximating 1° Engler. Schulz's formula (*Chem. Rev.*, 1900, 297) gives a fairly close approximation to the truth over a narrow range of viscosities, but outside this range the error is large, and the calculations in any case are very tedious. It is shown that no general formula to cover mixtures of hydrocarbons with fatty oils is possible, since mixtures of widely different viscosity result when light mineral oil is mixed with a fatty oil or with a heavier mineral oil identical in viscosity with the fatty oil. For mineral oil mixtures, however, a satisfactory method has been worked out. The method is not claimed to be exact, since it ignores the fact that two mineral oils of identical viscosity but different origin may differ notably in specific gravity, which affects the Engler readings. For many technical purposes, however, the errors of the method are said to be negligible.

The method is based on a curve connecting the observed Engler numbers of a series of mixtures of a light and heavy oil with the percentage of the heavy component. The oils used were American spindle oil (2·5° Engler and sp. gr. 0·868) and a clear, refined Russian oil of 67·4° Engler and sp. gr. 0·912. Any oil of intermediate viscosity can then be, and for the purpose of this method is, regarded as a mixture of the above two oils. To calculate the viscosity of a mixture in equal proportions of two oils of 10° and 40° Engler, one proceeds as follows: The curve shows that the former may be regarded as a mixture of 55 parts of the author's heavy oil (67·4°) with 45 parts of the light oil (2·5°), whilst the latter may be regarded as a mixture of 90 parts of the heavy oil with 10 parts of the light oil. The whole mixture may therefore be regarded as a mixture of 72·5 parts of the heavy oil with 27·5 parts of the light oil, and the curve shows that such a mixture has an Engler value of 19°. The curve needs to be drawn to a large scale for accurate work, and the following table derived from it will be of more service than a reduced facsimile of the curve. The volume percentages are the percentage of oil of 67·4° Engler in a mixture of such oil with light oil of 2·5° Engler, such that the viscosity of the mixture is that given in the first column.

Viscosity, ° E.	Volume, per Cent.	Viscosity, ° E.	Volume, per Cent.	Viscosity, ° E.	Volume, per Cent.	Viscosity, ° E.	Volume, per Cent.
2·5	0·00	5·0	32·07	21·0	74·93	46·0	92·80
2·6	2·50	5·1	32·79	22·0	76·05	47·0	93·20
2·7	4·80	5·2	33·50	23·0	77·11	48·0	93·60
2·8	6·80	5·3	34·20	24·0	78·11	49·0	94·00
2·9	8·67	5·4	34·88	25·0	79·07	50·0	94·35
3·0	10·33	5·5	35·53	26·0	79·99	51·0	94·70
3·1	12·00	5·6	36·19	27·0	80·86	52·0	95·05
3·2	13·43	5·7	36·81	28·0	81·70	53·0	95·40
3·3	14·86	5·8	37·41	29·0	82·51	54·0	95·75
3·4	16·25	5·9	38·00	30·0	83·29	55·0	96·10
3·5	17·50	6·0	38·59	31·0	84·04	56·0	96·45
3·6	18·75	7·0	43·68	32·0	84·77	57·0	96·80
3·7	20·00	8·0	47·96	33·0	85·47	58·0	97·15
3·8	21·11	9·0	51·62	34·0	86·16	59·0	97·50
3·9	22·22	10·0	54·82	35·0	86·82	60·0	97·80
4·0	23·30	11·0	57·69	36·0	87·46	61·0	98·10
4·1	24·30	12·0	60·24	37·0	88·09	62·0	98·40
4·2	25·30	13·0	62·52	38·0	88·70	63·0	98·70
4·3	26·27	14·0	64·58	39·0	89·29	64·0	99·00
4·4	27·18	15·0	66·44	40·0	89·87	65·0	99·30
4·5	28·08	16·0	68·13	41·0	90·42	66·0	99·60
4·6	28·92	17·0	69·68	42·0	90·92	67·0	99·90
4·7	29·75	18·0	71·14	43·0	91·42	67·4	100·00
4·8	30·54	19·0	72·48	44·0	91·90		
4·9	31·31	20·0	73·74	45·0	92·40		

G. C. J.

New Method for Determination of Hydrocyanic Acid and Alkali Cyanides. G. E. F. Lundell and J. A. Bridgman. (*J. Ind. and Eng. Chem.*, 1914, **6**, 554-556.)—The method consists in titrating an ammoniacal cyanide solution, containing a small quantity of dimethylglyoxime, with standard nickel ammonium sulphate solution, until a permanent red precipitate is produced ($Ni = 4CN$). The solution to be titrated is most conveniently about 50 c.c. in bulk, but the accuracy of the method is but slightly affected if it is necessary to take more in order to get a large burette reading. At least 1 c.c. concentrated ammonia should be present; 5 c.c. is harmless, but higher concentrations should be avoided. If the nickel content of the nickel ammonium sulphate is not known accurately, it is determined by precipitation with dimethylglyoxime. In the titration of cyanide about 0·5 c.c. (not more) of a 1 per cent. alcoholic solution of dimethylglyoxime is used. With pure alkali cyanides the results agree with those obtained by the gravimetric method or Liebig's method; with commercial samples the new method gives more accurate results, as it is unaffected by chlorides or thiocyanates in any concentration. In presence of twice as much ferrocyanide as cyanide the results are 2 to 3 per cent. too high, and in presence of twenty times as much carbonate as cyanide 0·2 per cent. too high; but the proportions of these impurities in commercial cyanides are without measurable influence on the results. Applied to electroplating solutions of gold or silver, the new method, like Liebig's method, measures only the "free" alkali cyanide, not combined as double cyanide. This is the figure usually required. The method is not applicable to zinc baths, as the end-point is reached when the whole of the free cyanide and about one-third of the combined alkali cyanide have reacted. With zinc baths, Liebig's method gives the total cyanide, as does the method of Hannay, whether applied to baths of zinc, silver, or gold.

G. C. J.

Melting and Solidifying Points of Mixtures of Fatty Acids and Use of these Points to Determine Composition of such Mixtures. E. Twitchell. (*J. Ind. and Eng. Chem.*, 1914, **6**, 564-569.)—It is known (Heintz, *Pogg. Annalen*, 1854, **92**, 588; also Ostwald, *Lehrbuch der allgemeinen Chemie.*, 2te Aufl., **1**, 1017) that when, to any of the higher fatty acids, any other of the higher fatty acids is added in amount up to 20 per cent., the depression of the melting-point is proportional to the amount of the added acid, and almost independent of its kind. Ostwald explains this by the law of equal depression of the freezing-point for equal molecular proportions, an explanation which assumes that the different fatty acids concerned have identical molecular weights. This, of course, is not true, but the molecular weights of the predominant fatty acids of most natural fats are all high, and sufficiently alike for the purposes of the calculations described in this paper.

The research now described was undertaken with the idea of reversing the usual process, and instead of using the lowering of the freezing-point to calculate molecular weights, to consider the molecular weights as known, and estimate the quantity of dissolved fatty acid from the lowering of the freezing or melting point. Since the solidifying-point can be determined with greater exactness than the melting-point, the solidifying-point of known mixtures was first investigated; but it was found that the depression of the solidifying-point was by no means independent of the nature of the second fatty acid. Melting-points were therefore determined by a modification of the capillary-tube method, under somewhat rigid conditions, described in the paper, and Heintz's observation confirmed that the depression of the melting-point is proportional to the amount of the second acid and substantially independent of its nature. For example, a nearly pure stearic acid had a m.-pt. of 69·30° C.; a mixture of 9 parts of this acid with 1 part of palmitic, oleic, or behenic acid melted at 67·25° C. (depression 2·05); and a mixture of 8 parts stearic acid with 2 of palmitic, oleic, or behenic acid, or 1 of oleic and 1 of palmitic acid melted at 65·25° C. (depression 4·05). Similarly the m.-pt. of palmitic acid (62·44° C.) was depressed 2·05° C. by mixing with one-ninth its weight of stearic, oleic, or behenic acid, and 4·16° C. by mixing with one-quarter its weight of these acids. The m.-pt. of behenic acid (79·99° C.) was depressed 4·33° C. when 2 parts of stearic, palmitic, or oleic acid were added to 8 parts of behenic acid. In the analysis of mixtures of fatty acids the above data are used as follows: Twenty parts of the mixture are added to 80 parts of palmitic, stearic, behenic, etc., acids, and the melting-point of the mixture determined. For example, 20 parts of cottonseed-oil fatty acids with 80 of palmitic acid give a mixture melting at 59·26° C. This is a depression of 3·18° C., and corresponds to a mixture of 84·6 parts palmitic acid with 15·4 parts other acids. The 20 parts of cottonseed oil fatty acids therefore contain 4·6 parts palmitic acid, or 23 per cent. An extension of the method consists in separating the solid fatty acids from a mixture and examining them as already described, and also in hydrogenating the original fatty acids by the method of Sebatier and Senderens, and examining the hardened product by the melting-point method. For example, the fatty acids from cottonseed oil, examined directly as described, appear to contain nearly 3 per cent. of stearic acid; but the solid fatty acids, in which the stearic acid should be concentrated, show no higher percentage, and the apparent content of stearic acid is probably due to experimental error. The hydrogenated fatty acids when mixed with pure stearic acid depress the melting-point so little that the hydrogenated fatty acids must contain about 71 per cent. of stearic acid, corresponding to 71 per cent. of unsaturated acids with 18 carbon atoms in the original mixture before hydrogenation, the increase in weight due to hydrogenation being negligible in work of this kind. The mixed fatty acids contain no more palmitic acid after hydrogenation than were present originally, so that unsaturated acids with 16 carbon atoms must be absent. A similar analysis of menhaden-oil fatty acids is given, the results being more complex and less definite, as menhaden-oil contains nearly 20 per cent. of unsaturated acids, other than acids with 16, 18, and 22 carbon atoms.

G. C. J.

Estimation of Carbon in Soils and Soil Extracts. J. W. Ames and E. W. Gaither. (*J. Ind. and Eng. Chem.*, 1914, **6**, 561-564.)—The method of estimating total carbon in soils by oxidation with a mixture of chromic and sulphuric acids has been reported on adversely by Warrington and Peake (*J. Chem. Soc.*, 1880, **37**, 617), and by Cameron and Breazeale (*J. Amer. Chem. Soc.*, 1904, **26**, 29). Hall and Miller (*J. Chem. Soc.*, 1906, **89**, 595) obtained satisfactory results, but only by passing the products of combustion over heated copper oxide. The present authors find the method exact, and Hall and Miller's modification unnecessary, provided the chromic acid is added first and followed by a sufficient amount of concentrated sulphuric acid. They treat 1 to 3 grms. soil in a short-necked Kjeldahl flask with 3 to 4 grms. chromic acid dissolved in 10 c.c. of water, followed by 50 c.c. of sulphuric acid. Air free from carbon dioxide is drawn through the mixture, which is boiled, and the escaping gases pass through a reflux condenser and then to an absorption apparatus containing 25 c.c. of 4 per cent. sodium hydroxide. The carbon dioxide is determined by Brown and Escombe's double titration method (*cf.* Amos, *J. Agr. Sci.*, 1906, **1**, 322), a modification of Amos' apparatus permitting the use of the small volume of alkali solution stated. The method is applicable without important modification to 1 per cent. hydrochloric acid extracts and 4 per cent. ammonia extracts of soils. Of the latter, 50 c.c., without previous concentration, is treated as described. Hydrochloric acid extracts (50 c.c.) are evaporated to about 30 c.c. under reduced pressure and then treated as described, 4 c.c. of 10 per cent. thiosulphate being added to the alkaline liquid before titration to prevent the destruction of the indicators by traces of chlorine and chromyl chloride. Suitable apparatus, making the method quite a rapid one when applied to routine work, is figured in the text.

G. C. J.

Determination of Lime Requirements of Soil. H. B. Hutchinson and K. MacLennan. (*Chem. News*, 1914, **110**, 61-62.)—The first part of the paper consists of a review of earlier methods, each of which is said to be unsatisfactory, for reasons which are briefly stated. The following method, the results of which have been correlated with those of pot and field experiments, is recommended: A standard solution of calcium bicarbonate of approximately $\frac{N}{50}$ strength is made by suspending excess of calcium carbonate in water contained in a "Sparklet" siphon, and subsequently saturating the water with carbon dioxide derived from one of the bulbs of compressed gas supplied with these machines. The solution is diluted with about one-third its bulk of water, filtered, and its exact titre determined. A weighed quantity of soil (10 to 20 grms.) is placed in a bottle of 500 to 1,000 c.c. capacity, together with a measured portion (200 to 300 c.c.) of the standard solution, and the whole agitated in a shaking machine for three hours. The mixture is then filtered, and 100 or 150 c.c. of the filtrate is titrated with $\frac{N}{10}$ acid, using methyl-orange as indicator. From the difference between the result of this titration and the acid required by an equal volume of the standard solution, the minimum lime requirement of the soil is calculated. Plant experiments showed that, other things being equal, yields increase as the acidity of the soil is decreased, right up to the "neutral point" as determined above, and that further additions of lime are without effect.

G. C. G.

Printing Inks and their Analysis. J. B. Tuttle and W. H. Smith. (*J. Ind. and Eng. Chem.*, 1914, **6**, 659-660.)—A synopsis is given of two papers on this subject, which will be published shortly by the United States Bureau of Standards. The first paper deals with the analysis of the inks, the four types investigated being web-press, flat-bed, job, and half-tone inks. The oil is best separated from the pigment by dissolving the ink in benzene or ether, and submitting the mixture to centrifugal action; after the evaporation of the solvent, the oil fraction is then examined for unsaponifiable oils, resin, hard gums, and linseed oil. In the case of black inks, the pigment is ignited, and the ash tested for driers, ferric oxide, etc. Aniline dyes, if present, are separated by extraction with alcohol or other suitable solvent. The coloured inks require a preliminary examination to ascertain the nature of the pigment used. Some of the mineral pigments, such as vermilion and chrome green, may be estimated quantitatively; if the colouring matter consists of aniline dyes or lakes, it will usually be found sufficient to determine whether or not these colours are fast to light. Two tests for this purpose are given. Results of analyses are recorded, and a brief description is given of the relation of the laboratory test to the practical tests which may be made by the printer. The second paper gives information on the general subject of printing inks, a description of the materials used in the manufacture of the inks, and the relation between the inks and the paper. W. P. S.

Preparation of Dry Starch, Soluble in Cold Water, for Use as an Indicator. R. M. Chapin. (*J. Ind. and Eng. Chem.*, 1914, **6**, 649-650.)—Four hundred grms. of potato starch are mixed in a flask with 2,300 c.c. of water and 80 c.c. of $\frac{N}{1}$ hydrochloric acid, and the flask is then placed in a boiling-water bath. The neck of the flask should be inclined at an angle of about 45°, and is rotated until the starch begins to liquefy and the paste falls away from the sides of the flask; the heating is continued, with occasional shaking, for about one and a half hours, or until all lumps of gelatinised starch have disappeared. The mixture is now cooled to 50° C., rendered alkaline with ammonia (using methyl-orange as indicator), 800 c.c. of 95 per cent. alcohol are added, and the whole is strained through moderately coarse muslin. The liquid, while still at a temperature of about 45° C., is then run through a number of fine jets into 4 litres of 95 per cent. alcohol, the latter being stirred continuously. After standing for forty-eight hours, the greater part of the alcohol is decanted, the starch is transferred to a filter and washed with alcohol, until the alcohol passing through the mass has a specific gravity indicating a strength of 90 per cent. During the washing the starch should be stirred to prevent the formation of clumps or fissures. The starch is then drained by suction, and spread out to dry in a moderately warm place. The resulting product is fine white voluminous powder which dissolves when thrown into cold water; if it is moistened with a small quantity of water, it forms a gummy mass, which dissolves very slowly.

W. P. S.

Chemical Valuation of Wood for Pulp. M. L. Griffin. (*J. Ind. and Eng. Chem.*, 1914, **6**, 560-561.)—In a previous paper (*J. Ind. and Eng. Chem.*, 1914, **6**, 163) the author showed the inadequacy of the cord measure as a unit for valuing pulp wood. As a better standard he advocated a solid unit—the cubic foot—and illustrated how such a unit could be adapted in practice without inconvenience. A unit of weight as a still more trustworthy basis was also advocated.

There remains the most rational basis of all—namely, valuation according to the amount of fibre that wood will yield. The following method is recommended: Sticks to be tested are quarter sawn, and the sample obtained by planing thin shavings from one of the sawn faces. The shavings (3 grms.) are dried at 100° C., boiled for thirty minutes with 1 per cent. sodium hydroxide, and the mass well washed and squeezed, and placed under a bell-glass into which chlorine is led. Complete conversion of the lignone usually follows within one hour. The mass is then washed free from hydrochloric acid and impurities, placed in 2 per cent. sodium sulphite solution containing 0·2 per cent. of sodium hydroxide, and boiled for five minutes. The fibre is washed with hot water, and retreated, if necessary, with alkaline sulphite solution. It is then dried and weighed. Experiment shows that there is no necessary connection between fibre content and specific gravity or density of annual ring growth. Incipient decay is usually correlated with low specific gravity and low fibre content. G. C. J.

INORGANIC ANALYSIS.

Velocity of Solution of Blende and Galena in Dilute Sulphuric Acid. F. Rosenkränzer. (*Zeitsch. anorg. Chem.*, 1914, **87**, 319-334.)—The velocities were measured by the method of Mecklenburg and Rosenkränzer (ANALYST, 1914, 325) which depends on the transformation of the dissolved hydrogen sulphide into methylene blue, which is then estimated colorimetrically. It is found that for a particular mineral the velocity is directly proportional to the acid concentration, to the time of reaction, and to the mass of mineral present, provided this be in an equally fine state of division in all experiments. When the fineness is varied, the velocity varies inversely as the square of the mean diameter of the particles—that is to say, it is directly proportional to the total surface of the particles. The influence of temperature is shown in the following formula: $y = k.e^{a\theta}$, where y is the amount of hydrogen sulphide evolved, e is the base of natural logarithms, θ is the temperature in degrees C., and k and a are constants, a having a value closely approximating 0·043 for both blende and galena. The value of k in the above formula depends not only on the acid concentration, time of reaction, mass and state of division of the sulphide, but on the nature of the latter, and is more than twenty times as great for blende as for galena.

According to the views of Noyes and Whitney, and of Nernst and Brunner and others, on the velocity of heterogeneous reactions, the reaction velocity under parallel conditions might have been expected to be identical for blende and galena. The bearing of the facts now brought forward is discussed in relation to Noyes and Whitney's generalisation, to which other apparent exceptions have been found. In the particular reactions studied by the author, velocity of diffusion, regarded by Noyes and Whitney as the predominant factor in the velocity of heterogeneous reactions, cannot have an important rôle, since the results are not measurably different when the mixtures are continuously and vigorously agitated, or merely stirred occasionally. G. C. J.

Method for Rapid Quantitative Analysis of Bronze and Brass. R. E. Lee, J. P. Trickey, and W. H. Fegely. (*J. Ind. and Eng. Chem.*, 1914, **6**, 556-560.)—For the analysis of lead-copper alloys containing 5 to 6 per cent. of tin and smaller proportions of zinc, antimony, and iron, the authors found existing rapid methods insufficiently accurate even for purposes of works control, and the exact methods too slow. The following scheme was worked out and found sufficiently accurate and rapid, the maximum errors recorded being 0·15 per cent. on the alloy, and the average error is much smaller. Separate portions are taken for each determination. Lead is determined gravimetrically, 0·5 grm. or more of the alloy being covered with 12 c.c. of 30 per cent. tartaric acid solution, and dissolved by heating with 4 c.c. nitric acid. To the cooled solution 10 c.c. sulphuric acid is added, and the mixture heated sufficiently to expel nitrous fumes, but not to decompose tartaric acid. After dilution with 100 c.c. water and 75 c.c. alcohol, the lead sulphate is filtered off on a Gooch crucible, washed with dilute sulphuric acid, then with alcohol, dried on the hot-plate, and finally ignited to dull redness. With alloys poor in lead, more is taken for analysis, and the solution must be correspondingly diluted before filtration, to prevent occlusion of copper. Copper is determined by a slight modification of Low's method (*J. Amer. Chem. Soc.*, 1902, **24**, 1082), small amounts of ferric iron being prevented from interfering by adding ammonium fluoride before the final titration; antimony or arsenic, if present, must of course be oxidised by bromine, and the latter expelled by heating, before proceeding with the titration. Low's iodimetric method for tin is said to fail in presence of much copper. In consequence, tin (with any antimony) is estimated gravimetrically, 0·5 grm. alloy being decomposed with 5 to 10 c.c. of dilute (1 : 1) nitric acid, the mixture evaporated to a paste, boiled for some minutes with 15 c.c. dilute nitric acid, diluted to 200 c.c., filtered through a Gooch crucible, and the precipitate washed with dilute acid, then with hot water, dried on the hot-plate, and finally ignited at a red heat. The precipitate includes any phosphorus, and generally occludes some copper and lead, but the error due to the latter cause is small when the dried residue is boiled with nitric acid and the solution largely diluted before filtration as described. Antimony is estimated by a modification of Low's method, the novelty consisting in dissolving 0·5 grm. or more of the alloy in 25 c.c. sulphuric acid, boiled in a Kjeldahl flask, since alloys of high copper content resist solution when treated as directed by Low. In absence of iron, zinc is estimated by the ferrocyanide method as follows: The alloy (0·5 grm.) is dissolved in dilute (1 : 1) nitric acid, and the solution evaporated to dryness. The residue is taken up in 10 c.c. hydrochloric acid and 100 c.c. water, and lead, copper, and tin are precipitated by hydrogen sulphide at 70° C. The mixture is filtered through asbestos supported on a Buchner funnel, and the filtrate, after dilution to 200 c.c. and warming to 70° C., is titrated with ferrocyanide in the usual manner, and the result corrected by means of a blank experiment made under substantially identical conditions. When iron is present, its estimation is combined with that of zinc. The filtrate from the sulphides of copper, etc., is boiled with nitric acid to oxidise the iron, which is then precipitated as hydroxide by addition of ammonium chloride and the least possible excess of ammonia to the boiling solution.

G. C. J.

Estimation of Chromium and Manganese in Iron and Steel. F. C. T. Daniels. (*J. Ind. and Eng. Chem.*, 1914, **6**, 658-659.)—Chromium is estimated by the following modification of the persulphate method for manganese: The sample (1 grm.) is dissolved in 100 c.c. of hot dilute (sp. gr. 1·135) nitric acid, and when solution is complete, nitrous fumes are expelled by boiling. In presence of much combined carbon, it is well to destroy this by addition of a few crystals of ammonium persulphate and momentary boiling. To the unfiltered solution, which should not measure less than 75 c.c., 75 c.c. of 0·2 per cent. silver nitrate solution and 5 grms. of ammonium persulphate are added. The mixture is heated to boiling, boiled for one minute, and dilute hydrochloric acid is added drop by drop until the permanganate colour is completely destroyed. After boiling for one minute longer, the solution is cooled, mixed with a measured volume of $\frac{N}{10}$ ferrous solution, more than sufficient to reduce the chromate present, and the excess of ferrous solution is then titrated with $\frac{N}{10}$ permanganate.

Manganese and chromium are estimated together by proceeding as already described, but after the addition of persulphate, only just heating to boiling, then cooling, adding a measured volume of $\frac{N}{10}$ ferrous solution. more than sufficient to reduce the permanganate and chromate present, and then titrating the excess of ferrous solution with permanganate. Manganese is then estimated by difference. The method is best controlled by check essays of a standard steel of approximately similar composition to the samples analysed. G. C. J.

Effect of Ferric Salts and Nitrites on the o-Tolidine and Starch Iodide Tests for Free Chlorine. J. W. Ellms and S. J. Hauser. (*J. Ind. and Eng. Chem.*, 1914, 6, 553-554.)—In a previous paper (ANALYST, 1914, 46) the authors have suggested the use of a hydrochloric acid solution of *o*-tolidine in testing for very small quantities of free chlorine or hypochlorites in water. As the colour changes on which the method depends are due to the oxidation of the *o*-tolidine, any oxidising agent in sufficient concentration and under proper conditions might be expected to interfere. In practice, the only interfering substance likely to be present in a satisfactory drinking water is ferric iron, of which at least 1 part per million must be present for the interference to be appreciable. One part of nitrite nitrogen in ten millions produces a just appreciable effect. Ferric iron and nitrites interfere with the starch iodide method at least as much as, if not more than, with the *o*-tolidine method. G. C. J.

Electrolytic Separation of Nickel. C. W. Bennett, H. C. Kenny, and R. P. Dugliss. (*J. Phys. Chem.*, 1914, **18**, 373-384; through *Chem. Zentralbl.*, 1914, II., 120.)—A stationary electrode gives a much better yield of nickel than does a rotating electrode. From a solution containing 7 grms. of nickel ammonium sulphate and 1 grm. of nickel chloride in 100 c.c. of water, 75 per cent. of the nickel was deposited on a rotating electrode, whilst over 90 per cent. was obtained from a similar solution when a stationary electrode was employed; the current used was 1·5 ampère per sq. dm. On increasing the current to 3 ampères, 96 per cent. of the nickel was obtained on the stationary electrode, and this yield was improved by the addition of ammonia to the solution. A smaller quantity of iron is deposited with the nickel on a stationary electrode than is the case with a rotating electrode.

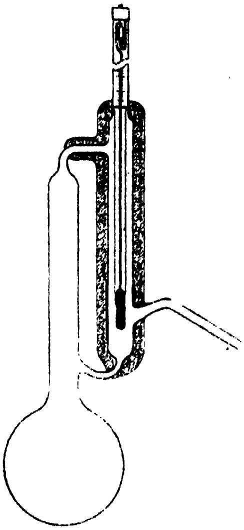
W. P. S.

Extraction of Radium from Australian Ores. S. Radcliff. (*British Association Report*, 1914, No. 139.)—A short account was given of all known occurrences of radio-active minerals in Australia. The methods now in use at the Sydney works of the Radium Hill Company for the extractium of radium from the complex ore found at Olary, near Broken Hill, were described. The results of an examination of the various radio-active precipitates separated in the course of the works operations were given, together with the methods employed in working up preparations of ionium and actinium.

Estimation of Silicic Acids. G. Tschermak. (*Zeitsch. anorg. Chem.*, 1914, **87**, 300-318.)—Some years ago (*Zeitsch. physikal. Chem.*, 1905, **53**, 349) the author described a method by which he claimed that many natural silicates could be referred to the type to which they truly corresponded, whether orthosilicates, metasilicates, or more complex compounds. The method consisted essentially in decomposing the silicates with acid, and then studying the rate at which the separated hydrogels lose water under constant conditions as regards temperature, pressure, ventilation, etc. The rate of loss of weight, rapid at first, falls slowly until a certain point—believed by the author to correspond to the loss of all mechanically held moisture—is reached. There is then a sudden marked fall in the rate of loss of weight, followed by a further gradual fall until—for any particular set of conditions—the rate becomes negligibly small. The material is finally dehydrated completely by ignition, and the moisture content at the time when the break in the dehydration curve occurred is calculated. If the moisture content corresponds to the formula $SiO_2.2H_2O$, the author regards this as evidence that the original silicate was an orthosilicate, and so on. Van Bemmelen (*Zeitsch. anorg. Chem.*, 1908, **59**, 225; *Chem. Weekblad*, 1908, **5**, 567) expressed the view that the hydrogels obtained from the silicates were colloidal adsorption compounds of indefinite composition, and that the changes in the rate of dehydration were probably due to changes in the mechanical structure of the hydrogels brought about by the gradual removal of water. The author has already (*Zeitsch. anorg. Chem.*, 1909, **63**, 230) met the criticisms of Van Bemmelen, who adduced no experimental evidence in support of his thesis. More recently Mügge (*Centralbl. f. Min.*, 1908, **129**, 325), and others have published data which they consider undermine the whole basis of the author's method. These results are now examined in detail, and it is shown that in some cases the periods of observation were so unfortunately chosen that no conclusion can validly be drawn from the experiments, whilst other series of results, when analysed, confirm in a remarkable manner the author's own results and conclusions. G. C. J.

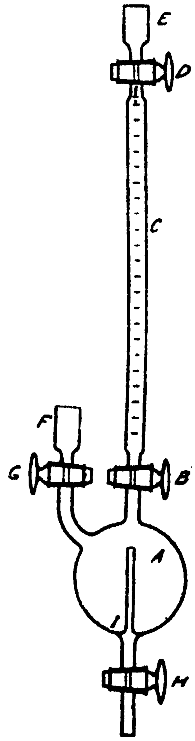
APPARATUS, ETC.

Still for Exact Measurement of Boiling-Point during Fractional Distillation. T. W. Richards and F. Barry. (*J. Amer. Chem. Soc.*, 1914, **36**, 1787-1791.)—Of the two modifications of this still figured in the paper, only one is reproduced here. The illustration is nearly self-explanatory. The Beckmann thermometer need not be fitted to the side-tube by a ground joint, except when dealing with hygroscopic liquids; the side-tube is constricted so that the thermometer passes within as far as the zero mark, and liquid soon condenses on the joint and makes a sufficient seal. The shaded portion represents asbestos, and the solid black lines right and left of the thermometer bulb represent tin foil introduced to minimise radiation. The rest of the apparatus is in one piece of glass, and of any convenient dimensions. The U-tube below the thermometer bulb for the return of condensed liquid to the flask should be of small diameter, and only just sufficiently inflected to serve as a trap, so that the dead space which can hold and thereby waste liquid may be as small as possible. With this apparatus 500 c.c. of good commercial toluene, with a b.-pt. range of rather more than 1° C., yielded, as the result of six fractionations, 130 c.c., boiling within 0·02° C., and from this, by two further fractionations, 80 c.c., boiling within 0·01° C., was obtained. By immediate immersion of the warm dry bulb of the thermometer in steam, the difference between the boiling-point of toluene and that of water was determined within 0·01° C. This is the type of purpose for which the apparatus was designed. In another form of the apparatus, figured in the paper, the Beckmann thermometer is replaced by a ground stopper, terminating below in a hook from which a small Auschütz thermometer can be hung. With this arrangement, mica windows must be provided in the asbestos mantle to make possible the reading of the thermometer. G. C. J.



Pitot-Tubes for Measurement of Gas Velocities. A. M. Fairlie. (*J. Ind. and Eng. Chem.*, 1914, **6**, 583-585.)—The author points out several serious misprints in the important paper of Rowse (*J. Amer. Soc. Mech. Eng.*, 1913, September) and in *Bulletin* **35** of the American Blower Company, whose standard-tube Rowse found to be the most accurate form available. With this tube and Rowse's data, gas velocities may be measured accurately within 1 per cent. in a 12-inch pipe, provided there is a straight run of 20 to 38 feet. Rowse has shown that there is an approximate relation between the velocity head at the centre of a pipe and the mean velocity head, but this relation is only known accurately for 12-inch pipes. The general proposition being established, however, and the requirements of a really trustworthy Pitot-tube being now known, further research is justified, and the author indicates the lines along which it should proceed. To establish the relation between velocity head at centre of pipes with mean velocity head for various sizes of pipe, at least 20 diameters in length, is a straightforward, if tedious, matter, the determination of the relation (if any) in the case of shorter pipes a more difficult but not less important one. It is pointed out that methods depending on the measurement of an average velocity head by a single reading of an instrument with many openings distributed across the diameter of the pipe, such that each opening represents an annulus of equal area, must fail, because the velocity varies as the root of the velocity head. To obtain a correct result, separate readings for each annulus would have to be made, the roots extracted and averaged, and the final result squared. G. C. J.

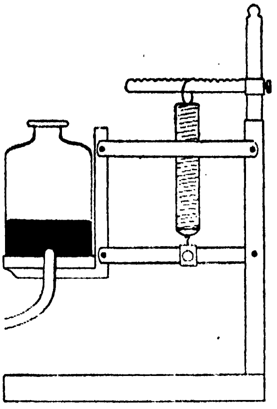
New Apparatus for Estimation of Hydrogen Sulphide in Water. Part I. G. B. Frankforter. (*J. Ind. and Eng. Chem.*, 1914, **6**, 676-677.)—Of two pieces of apparatus figured in the original, only that which is said to be the more satisfactory is reproduced here. To take a sample, *B* is closed, *G* and *H* are opened, and the apparatus plunged in the water. When *A* is full, the apparatus is raised out of the water and held in a strictly vertical position until water no longer flows from *H*. *G* and *H* are then closed. The exact amount of water enclosed in this way is determined once for all by trial, 250 c.c. being a convenient volume to aim at when constructing the apparatus. Starch solution is introduced through *F*, and standard iodine solution (1 c.c. = 1 c.c. H_2S at N.T.P.) is run in from the burette *C* until a permanent blue colour results. The estimation can be made within a minute of taking the sample, and should be completed as rapidly as possible. G. C. J.



Non-Uniformity of Drying-Oven Temperatures. L. H. Bailey. (*J. Ind. and Eng. Chem.*, 1914, **6**, 585.)—The increasing use of electrically heated ovens, and the reliance placed on them, has led the author to investigate the range of temperature in different parts of several such ovens, maintained approximately at 100° C. The best of them showed a variation of 10° C. on the upper shelf, 13° C. on the lower shelf, and 15° C. between the coolest spot on the lower shelf and the hottest on the upper. Gas-heated air ovens show about the same variations as electrically heated ones. In a large (15 inch cube) rectangular hot-water oven, the maximum variation on one shelf may be 3° C., and throughout the oven 4° C., but in small ovens (15 inch × 8 inch diameter) a uniform temperature of 100° C. is easily secured. G. C. J.

Bernadot's Vinometer. Fleissig. (*Schweiz. Apoth. Zeit.*, 1914, **52**, 265; through *Chem. Zentralbl.*, 1914, II., 189.)—The wine is poured into a bell-shaped expansion at the top, and on inverting the instrument the liquid rises in a capillary tube to a point depending upon the amount of alcohol, the percentage of which may then be read directly upon a scale. Wines rich in alcohol must be diluted with an equal quantity of water. The apparatus is made by Werthemann, Botty and Co., Basel. C. A. M.

Manostat for Use in Gas Analysis. H. N. Gilbert. (*J. Ind. and Eng. Chem.*, 1914, **6**, 585-586.)—It is sometimes important to maintain a nearly constant pressure in a combustion pipette, and difficult to do this by hand. The apparatus figured accomplishes it automatically. The levelling bulb, in the form of a flat-bottomed bottle, is supported on the shelf shown, which is attached to the two hinged arms (horizontal in the figure), the lower arm carrying a sliding block attached to the lower end of the helical spring shown. By moving the point of support of the spring along the toothed arm, it can be so adjusted that the vertical distance through which the levelling bulb moves when the spring is stretched by the inflow of more mercury from the pipette is just equal to the height of the column of mercury leaving the pipette. The apparatus is made by Greiner and Friedrichs, and known as the "Gilbert Manostat." G. C. J.



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REPORTS.

Report of the Government Chemist upon the Work of the Government Laboratory for the Year ended March 31, 1914, with Appendices.

(Issued as a Parliamentary Paper [Cd. 7562], pp. 1-25.)—The number of samples examined in the course of the year shows an increase of 25,252 over that of the previous year, the totals for the two years being 234,754 and 209,502 respectively, exclusive of the samples dealt with at the Chemical Stations in the provinces. Including these samples the total amounts to 392,367.

The chemical work of the following Departments and other public bodies is now carried out in the Government Laboratory: Board of Customs and Excise, Admiralty, Board of Agriculture and Fisheries, Department of Agriculture and Technical Instruction for Ireland, Colonial Office, Crown Agents for the Colonies, Foreign Office, Home Office, India Office, Board of Inland Revenue, Local Government Board, Post Office, Public Record Office, Stationery Office, Board of Trade, Trinity House, War Office, and Office of Works (London and Dublin).

Customs and Excise.—The work for this Department consists mainly in the examination of samples in connection with the assessment of duty and drawback, or with regulations regarding the manufacture and sale of dutiable articles. *Beer.*—The number of samples examined in connection with the beer duty was 51,417. *Materials used in Brewing.*—Seven hundred and thirty-six samples were analysed, including 447 samples of malt, corn, brewing sugars, and exhausted grains, and 289 of yeast foods, finings, etc. Of 91 samples of finished beer examined for the presence of saccharin, saponin, and other prohibited ingredients, only 2 samples were found to contain saponin. *Worts, or Beer in the Unfinished Condition.*—Out of 12,510 samples examined, the original gravity was declared by the brewers at less than the true gravity in 1,713 cases, the percentage of under-declarations being 16·7 as compared with 17·2 per cent. in the previous year. *Beer as retailed.*—The beer was found to have been diluted in 557 samples out of a total of 7,986; many of the samples were diluted to the extent of 6 to 10, and in some cases 12, gallons of water to the barrel of 36 gallons. *Herb Beers, Beer Substitutes, and other Temperance Beverages.*—The percentage of alcohol was estimated in 511 samples; in 401 samples the alcohol did not exceed the legal limit, 74 samples contained between 2 and 3 per cent. of proof spirit, 31 between 3 and 5 per cent., and 5, described as ginger-beer or herb-beer, from 5 to 7 per cent. *Examination of Beer and Brewing Materials for Arsenic.*—The total number of samples examined, including beer, worts, malt, sugar, and other materials, was 1,596; of these, only 7 were found to contain arsenic in excess of the limits laid down by the Royal Commission on Arsenical Poisoning. All the samples, 306, of malt and sugar tested were free from arsenic, and only one of the 1,143 samples of beer contained arsenic in excess of the limit, the amount in this case being $\frac{1}{36}$ grain per gallon. Six cases of excess were detected amongst the miscellaneous samples, the highest amount of arsenic found being $\frac{1}{8}$ grain per gallon. *Cider and Perry.*—Of 7 samples purchased as cider by the Customs and Excise officers, 4 were found to be artificially prepared liquids, and 8 samples purchased by inspectors of the Board of Agriculture and Fisheries were of a similar character. Two hundred and eighty-six samples of imported cider were examined; one proved to be factitious and another proved to be liable to duty as "wine." *Fusel Oil.*—This material, separated during the distillation of spirits in patent stills, is allowed to be delivered duty free, provided that it does not contain more than 15 per cent. of proof spirit; of 40 samples examined, 2 contained an excess of proof spirit. *Liqueur Chocolates.*—Twenty-five samples of these chocolates were analysed; 4 samples contained an amount of proof spirit ranging from 5 to 8 per cent., and in 3 other samples the amounts were 16·5, 18·0, and 33·1 per cent. respectively. The average amount of proof spirit found in 624 samples of imported chocolates was 4·3, the highest being 16·4 per cent. *Methylated Spirit.*—Nine hundred and sixty-one samples of wood naphtha were submitted for approval as to their suitability for denaturing spirit; 7 of these samples were rejected. All the samples of mineral naphtha, 224, submitted were approved. Only 32 samples of petroleum spirit, used in conjunction with methylated spirit in the manufacture of felt hats, were submitted, and there was a marked reduction in the quantity of this denatured spirit employed in the industry. *Wine.*—The samples numbered 98,802, the examination being mainly restricted to the estimation of the alcoholic strength. *Tobacco.*—The work in connection with tobacco consists chiefly in the examination of unmanufactured and manufactured import tobacco, tobacco manufactured in the country for home consumption, tobacco and commercial snuff for export, and offal tobacco. Manufactured tobacco must not contain more than 32 per cent. of water, and, where oil is used, the amount of this must not exceed 4 per cent. Moisture was estimated in

16,518 samples, and oil in 2,402 samples. The number of samples of snuff examined was 214, and of offal tobacco, 44,088. Seven samples of cut or cake tobacco were found to be adulterated with liquorice. Eighteen hundred and eighty-five samples of nicotine solutions for horticultural purposes, etc., were examined. *Sugar, Glucose, and Saccharin.*—The samples of sugar and articles containing sugar examined amounted to 42·644, the analyses being made for the assessment of duty. In relation to articles containing sugar, it may be mentioned that imported honey, although not liable to duty when genuine, is frequently adulterated with invert sugar. Of 75 samples analysed, 32 afforded evidence of the presence of added invert sugar, and were accordingly charged with duty. Owing to the heavy duty on saccharin, there is a great inducement to smuggle this article, and search has to be made for it in all preparations in which there is any probability of its occurrence; of 304 samples of condiments, dentifrices, and medicinal preparations tested, 105 were found to contain saccharin. *Tea.*—Nine thousand three hundred and three samples were received; 800 were condemned as containing sand, etc., and 74 were reported as being unfit for human consumption. *Coffee.*—If not mixed with chicory, coffee is entitled to drawback when exported; 2 samples out of 1,548 examined were found to contain chicory. The coffee substitutes analysed consisted of chicory, caramel, roasted cereals, and figs. *Cocoa and Chocolate.*—The examination of 3,847 samples of these preparations related to the estimation of cocoa, sugar, and cacao butter for the purpose of assessing duty and drawback. *Matches.*—The presence of white phosphorus was not detected in any of the 1,333 samples examined.

ADMIRALTY.—The substances examined included foods, metals, soaps, pottery, etc. Nearly all the samples of dairy produce were genuine; 1 sample of milk was deficient in fat; 1 sample described as butter consisted of margarine, and 9 samples of butter from naval hospitals contained boron preservative contrary to the specification. A sample of tinned crayfish contained 6 grains of tin and 1 grain of lead per pound.

BOARD OF AGRICULTURE AND FISHERIES.—The number of samples examined for this department, including those for the Board of Agriculture for Scotland and the Department of Agriculture and Technical Instruction for Ireland, was 3,545. *Imported Milk and Cream.*—Of the 83 samples of condensed sweetened and unsweetened milk, 70 were condensed whole milk and 13 condensed skimmed milk; 1 sample, marked "Condensed," was found to be impoverished whole milk, and this sample also contained boric acid. The percentage of fat in the condensed whole milk varied from 8·0 to 11·4. All the samples of condensed milk were free from preservatives. Eighty-nine samples of milk powder were analysed; 34 consisted of whole milk, 2 had been prepared from impoverished milk, and 51 from skimmed or separated milk. There was a large decrease in the quantity of milk imported in churns, and all the four samples taken were genuine. One sample of sterilised milk was slightly deficient in solids-not-fat, 7 were genuine, and all were free from preservatives. The quantity of fat in 98 samples of cream varied from 19 to 59·3 per cent.; 3 of the samples contained sugar, 1 added water, and another gelatine. Fifty-eight of the samples of cream contained hydrogen peroxide and 23 boric acid. *Imported Butter.*—One thousand two hundred and fifty-eight samples were taken; 355 contained boron preservative, and 148 added colouring matter. The quantity of water present varied from 10·68 to 14·57 per cent., but 1 sample of Italian butter contained 17·38 per cent. The Reichert-Wollny value fell below 24 in 18 cases (13 of these 18 samples were from Russia and Siberia), the lowest value found being below 20. *Margarine.*—All the 671 samples of imported margarine were free from excess of butter-fat, but 22 contained excess of water; paraffin-wax was not present in any of the samples. *Imported Cheese.*—Of the 72 samples examined, 1 contained foreign fat and several others had been prepared from skimmed milk. *Samples taken under the Butter and Margarine Act.*—Of 612 samples submitted, 38 samples of butter, 2 of milk-blended butter, and 5 of margarine contained excess of water. *Sheep-Dips.*—One hundred and nine samples of sheep-dips were received for examination in connection with applications by manufacturers for inclusion of their preparations in the official schedule of efficient dips. In 79 cases the samples were of a satisfactory nature, 18 were deficient in active ingredients at the dilution specified, and in 12 cases the formulæ required modification. Forty-two samples of approved dips, purchased in various parts of the country, were also analysed, and 11 of these were found not to have been prepared according to the formulæ previously submitted and approved. *Copper Sulphate.*—Forty-three samples consisted of commercially pure crystallised copper sulphate; 12 samples contained large proportions of iron sulphate, and 2 consisted almost wholly of powdered iron sulphate coloured with Prussian blue. *Apples.*—Apples containing a slight deposit on the skin near the stem and near the "eye" were found to be contaminated with traces of copper, probably due to spraying with Bordeaux mixture; arsenic was not present. *Feeding-Stuffs.*—Several samples were examined as to their purity or in connection with cases of illness in cattle. An oil which was stated to have caused illness in cattle was found to consist of genuine raw linseed oil. *Soils.*—Samples of soil were analysed in connection with an inquiry regarding causes of disease affecting apple-trees. Other samples of soil were taken from plots where afforestation experiments are being made. *Questions submitted for Report.*—Amongst many other matters the following were submitted: The use as a fertiliser of sodium nitrate containing perchlorate; methods of sealing samples; composition of milk powder; alleged use of sugar as a preservative in butter; the necessity for making a statement of the water-soluble phosphates in guano; sampling of calcium nitrate; use of preservatives in bacon curing; sale of honey substitutes; alleged use of milk powder in Devonshire cream; milk deficient in lactose; presence of gypsum in sharps; the relation between citric acid solubility and the availability of phosphates in slags.

FISHERIES DIVISION.—Thirty-eight samples of river water were analysed. These waters were believed to have caused injury to fish. In one case a dead salmon, taken from the same stream as one of the samples of water, was found to contain small quantities of copper and zinc. In another case the water discharged into the river contained iron, arsenic, and mineral acid. Samples of mud and silt from sea and river bottoms were also examined, and 4,314 samples of sea-water were analysed.

HOME OFFICE.—Most of the work done for this Department was in connection with various regulations and inquiries affecting the health of workers in factories. The dangers connected with the use of lead continue to receive a large amount of attention (*cf.* ANALYST, 1913, **38**, 538.) In 51 cases where lead-poisoning had occurred, it was found that almost all the lead in the glazes used was soluble in dilute acid. One glaze contained 70 per cent. of lead monoxide, of which practically the whole was soluble. The effect of gas-stoves on the air of factories was investigated.

LOCAL GOVERNMENT BOARD.—*British Wines and Cordials.*—In connection with an inquiry into the use of preservatives and colouring matters in the manufacture of British wines and cordials, the examination of a large number of samples of these beverages, together with samples of flavouring, colouring, and preservative substances, was carried out, and a full report was made to the Board. *Preservatives in Food.*—All the samples of milk and cream taken under the Food and Drugs Acts and the Public Health Acts were examined as to compliance with the Milk and Cream Regulations. The samples of whole milk imported in churns, of sterilised milk, sterilised cream, and condensed milk were free from preservatives with one exception. The preservatives found in "churn" cream during the year were boric acid and hydrogen peroxide, but since October, 1913, boric acid was not found in any of the samples, hydrogen peroxide having been used in all cases. The amount of boric acid found ranged from 0·12 to 0·49 per cent. Boric acid was the only preservative found in butters coming into the country; all the samples of butter from Denmark, Norway, and Sweden were free from boric acid, and only 2 of the samples from Russia contained this preservative. The percentage of samples from Australia containing boric acid was 29. *Old Age Pension Act.*—Nine documents referring to names and dates were submitted for examination on account of their suspicious appearance, and in 5 cases it was found that the writing was of recent date or that it had been tampered with recently.

PUBLIC RECORD OFFICE.—At the request of the Deputy Keeper of the Records, analyses were made of a series of medieval wax impressions dating from the thirteenth to the sixteenth century. Most of the seals were found to consist of mixtures of beeswax and resin. The wax in the case of two of the seals, dated respectively 1399 and 1423, possessed the character of East Indian rather than European beeswax. An impression of the Great Seal of 1350 consisted of pure beeswax, and the wax, although nearly six centuries old, corresponded exactly in its properties with wax of recent origin. The colouring matter of the seals was usually vermilion or verdigris.

BOARD OF TRADE.—The lime and lemon juice supplied to foreign-going ships is now examined regularly in the Government Laboratory; of 379 samples of raw lime-juice analysed, only one was rejected as being unsuitable. An investigation was made for the Board as to the suitability, from a hygienic point of view, of galvanised vessels as milk measures. It was found that fresh milk kept in a galvanised vessel for periods up to two hours dissolved no zinc, or only slight traces. Appreciable quantities of zinc were, however, dissolved when the milk was allowed to go sour in the vessel; at the end of twenty-four hours the milk contained 0·5 grain of zinc per pint. Traces of arsenic and antimony, present as impurities in the zinc coating of the vessel, were also found in the milk which had been kept for twenty-four hours in the vessel.

Office of Works, London.—The samples examined consisted largely of materials supplied by contractors for public service, and were of very varied character. Specimens from the "vitrified" fort of Finavon were examined for the Department to ascertain the nature of the flux, or binding material, which holds the stones of the walls of the fort together. The material differs in composition from the stones; it is similar to certain felspathic rocks, and has a fusing-point of about 2,000° F. Assuming the vitrification to have been brought about by heat, it would appear that the builders of the fort must have been acquainted with some special means, such as a wind furnace would supply, for obtaining high temperatures.

Sale of Food and Drugs Acts.—*Examination of Samples referred by Magistrates under the Sale of Food and Drugs Acts of* 1875 *and* 1899.—Seventy-three samples were received under the provisions of these Acts, as compared with 92 samples in the previous year. These included 42 samples of milk, 4 of whisky, 3 of jam, 2 each of butter, pepper, and rum, and 1 each of margarine, almond oil, coffee, dripping, gin, lard, jelly, and vinegar. In the case of 2 samples of milk in which decomposition had proceeded to an unusual degree, no certificate was issued. Six samples of milk forwarded could not be examined, the bottles having burst in transit. The results of the analysis at the laboratory differed from those of the Public Analyst in 8 cases out of 65, in respect of which certificates were issued; three of the disagreements related to milk samples. The following are the particulars of some of the more important cases: *Butter.*—In both cases the butter was found to contain excess of water, as alleged by the Public Analyst. *Whisky, Gin, Rum.*—Except in two cases, the results obtained confirmed those of the Public Analyst. *Jam.*—The samples were found to contain apple-pulp, as alleged. *Pepper.*—The pepper was sold as white pepper, but contained an excessive amount of husk; the results were in agreement with those of the Public Analyst. *Margarine.*—In this case the result did not confirm the allegation of the Public Analyst that the margarine contained an excess of butter-fat. *Almond Oil.*—It was alleged that the sample contained peach and apricot kernel oils, but no evidence of the presence of these oils could be obtained. *Dripping.*—Cottonseed oil was stated by the Public Analyst to be present, but none could be found. *Milk.*—In the 44 cases in which certificates were issued, the charges were as follows: In 20 cases, added water; 16, abstraction of fat; 4, added water and abstraction of fat; 4, presence of boric acid. In regard to the samples of coffee alleged to contain chicory, of lard alleged to contain beef or mutton stearine, of red currant jelly alleged to be mixed with gooseberry jelly, and of malt vinegar alleged to contain vinegar not derived from malt, the results agreed with those of the Public Analyst. Forty-nine other samples were also analysed in order to obtain data for the purpose of comparison.

Fertilisers and Feeding-Stuffs Act.—Twenty-four samples, consisting of 9 fertilisers and 15 feeding-stuffs, were submitted by the Board of Agriculture and Fisheries in connection with applications by local authorities for the consent of the Board to proceedings against the sellers. The fertilisers consisted of basic slag, potato and root manures, guano, fish manure, and nitrate of soda. The nitrate of soda contained a considerable amount of perchlorate, and the other fertilisers were deficient in phosphates, nitrogen, or potash. The feeding-stuffs consisted of cod-liver oil, poultry food, and various feeding-meals. Several of the meals were deficient in oil or in albuminoids; the cod-liver oil was in a rancid condition, and contained water. W. P. S.

Reports to the Local Government Board: I. On the Use of Proprietary Foods for Infant Feeding. F. J. H. Coutts. II. On the Analysis and Composition of Some Proprietary Foods for Infants. Julian L. Baker. (*Food Reports*, No. 20. 1914. Wyman and Sons, London. Pp. 83. Price 6d.)—These two reports have been prepared in response to numerous representations made both at the National Conference on Infant Mortality in 1906, and also by County Councils and others, that action should be taken to restrict the use of unsuitable infant's foods. With the assistance of Medical Officers of Health in various parts of the country, a large number of different infant's foods—106 in all—were obtained, and of these 29 representative samples were selected for detailed chemical analysis. It was considered preferable to have an exhaustive examination made of a comparatively small but representative number rather than a less complete examination of all, and at the same time to restrict this examination mainly to one particular class of foods, viz., those containing or supposed to contain starch in an unchanged or only slightly altered form.

It was decided that the selected samples should be examined to determine as accurately as possible:

(1) The presence, nature, and amount of any unaltered starch; (2) the extent to which conversion of starch took place when the food was prepared according to instructions on the labels; (3) the nature of the cereal from which the starch was derived; (4) the presence or absence of diastase in active form.

The proprietary foods may be divided into three groups:

(1) The widely advertised brands made by large manufacturers and sold by pharmacists and stores all over the country; (2) preparations manufactured on a smaller scale by pharmacists or others, and sold chiefly in one locality under the name of the person responsible for the production; (3) preparations manufactured on a large scale by wholesale firms and distributed to pharmacists, stores, etc., in packages marked with the name of the retailer. These are sometimes known as "own-name" brands.

Probably Group No. 2 is a comparatively insignificant class at the present day. It seems represented chiefly by preparations consisting of what is practically nothing more than baked wheat-flour, put up in packets and sold at a fancy price as "———'s Infant Food."

Group No. 3 probably forms a very large proportion of the total. In many cases in examining and comparing the packages it was obvious that a certain group of samples purporting to be entirely different brands were in all probability the product of one factory. Dr. Coutts states that he was able to compile a list of over ten manufacturers who prepare infant's foods for the trade, packing them ready for sale with the name and address of the pharmacist or dealer.

It is obvious that many of these retailers can have little knowledge of the article they are selling as their own proprietary infant food. They have absolutely no control over the quality of the materials used, or over the conditions under which it is manufactured, and considerable variations in composition may occur from time to time without their being in the least aware of them. Actual cases illustrating the probability of such changes occurring are given on pp. 15 to 16 of the Report.

Apart from special preparations of cow's milk, such as humanised milk, condensed milk and dried milk, the proprietary infant's foods may be conveniently grouped in the following five broad classes:

(1) Foods with a basis of dried cow's milk but mixed with flour. (2) Foods consisting mainly of flours, the starch of which is practically unaltered, or altered only by heating. (3) Foods consisting mainly of flours mixed with a proportion of malt flour or malt extract, but containing much unaltered starch which is not converted during the process of preparing the food for infants in accordance with the directions given on the package. (4) Foods containing flours, but also containing active diastase or pancreatic ferment, so that if the food is carefully prepared according to the directions on the package the starch is appreciably altered. (5) Foods manufactured from flours, the starch of which has been mainly or partially converted into soluble products during the course of manufacture.

The samples for analysis were chosen so as to give representations of all these five classes. No samples which appeared to consist merely of dried milk or dried milk *plus* milk products were included, as a separate report on dried milks is in course of preparation.

The twenty-nine samples examined by Mr. Baker may be arranged in the five classes as follows:

Class.	Numbers.	Total.
I.	54, 68, 73	3
II.	1, 3, 4, 7, 9, 13, 17, 28, 29, 31, 51, 58, 60	13
III.	14, 16, 20, 21, 26, 33, 44 ...	7
IV.	32, 56, 63, 83	4
V.	2, 36	2

In the case of the remainder of the 106 samples which were not subjected to detailed chemical analysis, a rough classification from statements on the labels or from other evidence indicated that probably over 70 per cent. of the articles sold as infant's foods belong to Classes II. and III. and contain material quantities of starch which remains practically unchanged when the food is prepared according to directions.

An examination of the tables of analytical results given by Mr. Baker shows that the chemical composition of the food investigated is in most cases very far removed from that of dried human milk. But as many of these foods are directed to be prepared with milk, the composition of the mixture of patent food and milk, when prepared according to the directions on the package with cow's milk of average quality, was calculated, and a table on p. 13 of the Report gives the result of these calculations as applied to the various samples when mixed as directed, figures for human milk and for cow's milk being added for comparison.

In these mixtures the protein is usually in considerable excess as compared with human milk, and approximates to the proportion in cow's milk. The fat is very deficient in some cases and rather low in many others. In many of the foods as prepared for use the amount of carbohydrate is proportionately much too great for the amount of fat and it is generally considered that such a disproportion is one of the causes of rickets.

Even when made with the full quantity of cow's milk some of these mixtures contain unchanged starch in very excessive proportions.

In the early days of food chemistry the gross chemical composition of food elements was apparently considered sufficient evidence of the suitability of the food. All proteins were looked upon as the same in constitution; one sugar was supposed to be able to replace perfectly any other, and, moreover, all carbohydrates were considered to be equally assimilable. Starch might therefore, according to this view, quite well replace sugar, and there was a strong tendency to regard carbohydrates as being capable also of replacing fats. As Dr. Coutts points out, nutrition is not such a simple chemical problem, but the persistence of these old views probably explains such statements as the following, which appears on the labels of several presumably different brands of infant's foods: "It contains all the nutritive elements of human milk—carbohydric, albuminous, and saline—in approximately the same proportion in which they exist in this secretion, *and is therefore capable of supplying an equivalent of natural nourishment.*"

Liebig's authority is invoked on several labels, especially those of foods claimed to be malted—"prepared on strictly scientific principles as laid down by the late famous *Professor Liebig*"; and it would appear that many of the firms manufacturing infant's foods at the present day base their views as to the suitability of the diet on the chemical pronouncements of Liebig.

Pp. 16 to 31 of Dr. Coutt's Report are devoted to a comprehensive discussion of the suitability of starch-containing foods for infant feeding, considered from a physiological standpoint, and practically all the more important literature of the subject comes under review. Dr. Coutts concludes that for practical purposes salivary digestion of starch in the ordinary infant is a negligible matter, and that although numerous observations have been recorded which show the possibility of pancreatic action, the mere presence in small amount in the infant organism of amyolytic secretions is not sufficient proof that starch is a suitable food for infants. Isolated cases, "starch babies," are undoubtedly met with, but the fact that idiosyncrasies of this kind exist does not justify the routine use of starchy foods for any or all babies. The results of clinical experience, moreover, give us positive proof that starch is as a rule harmful, and from an inquiry carried out by Dr. Howarth in Derby during the years 1900-1903, when the nature of the food given to 6,904 babies was ascertained, it would appear that the use of foods containing starch was responsible for an increased rate of infant mortality.

In the great majority of cases it is expressly directed that milk is to be used along with the food, and on close examination of the directions, it is obvious that, if these are properly followed, milk forms the main basis of the diet.

But there are many considerations which tend to induce the mother to omit altogether, or to reduce the quantity of milk, a practice which is encouraged by the fact that several brands of infant's foods go to the length of suggesting that milk is not necessary, by directing that the food can be made up with milk *or water.*

Dr. Coutts gives a selection of various laudatory statements taken from the labels of different brands, one of which (No. 33) is declared to be "*greatly superior* to a healthy mother's milk," although 73·6 per cent. of starch is present.

Several of the foods are declared to contain malt, and to be "self-digesting," but although there may be active diastase present, yet if the food is prepared according to directions, no material conversion of the starch takes place.

Health visitors frequently experience great difficulty in persuading mothers that the highly coloured statements in many advertisements about patent foods are not to be implicitly relied upon.

Public analysts have frequently reported on the results of analysis of these foods, and have commented strongly on the lack of correspondence between the analytical findings and the claims made in the advertisements, but as a rule local authorities have not felt justified in prosecuting the vendors.

Dr. Coutts closes his report by discussing the various suggestions which have been made from time to time as to the most effective administrative action, and points out that probably none of the suggested methods of exercising control over unsuitable infant's foods would be entirely satisfactory unless steps could be taken to prevent the mischievous effects of inaccurate titles or misleading statements in placards, advertisements, pamphlets, or directions for use accompanying infant's foods.

In Mr. Julian Baker's report the methods of analysis adopted are described at length, and complete analyses of twenty-nine representative infant's foods are given. The analysis of mixtures which may contain lactose, cane-sugar, dextrose, invert sugar, hydrolysed starch products and unconverted starch, is a lengthy and laborious matter. With a view to the accurate estimation of such small quantities of lactose as are present in many of these foods, a fermentation method was devised by the author and H. F. E. Hulton, an account of which has already appeared in the ANALYST (*cf.* J. L. Baker and H. F. E. Hulton, ANALYST, 1910, **35**, 512).

Unconverted starch was estimated by a polarimetric method (*cf.* Thorne and Jeffers, ANALYST, 1909, **34**, 332), the reliability of the method having been previously tested by analyses of pure starches.

From the difference between the amount of aqueous extract at 15·5° and 35° C., provided that active diastase is absent, some idea can be obtained as to the extent to which the starch has been altered or rendered partially soluble by heating or otherwise.

The constituents included under the term "hydrolysed starch products" comprise the mixture of maltose, malto-dextrins and dextrins obtained by the action of diastase on starch, all of which are soluble in water and are optically active. In some of the foods examined—*e.g.*, Nos, 2, 36, 54, and 68—there is present what is probably an evaporated starch or flour conversion, consisting of a mixture of carbohydrates obtained by the action of diastase on starch. As a result of this process, the starch is changed into a mixture of malto-dextrins and maltose. The amount of such products in a mixture cannot be determined by any simple direct method, but as a rule it can be arrived at from the specific gravity, copper-reducing power and optical activity of the filtered aqueous extract of the food, after allowance has been made for the other soluble bodies—*e.g.*, cane-sugar, lactose, proteins, etc.—which had been directly determined.

A word of warning is given as to the estimation of fat in these foods. Mere ether extraction is often insufficient to deal with the fat in some foods, more especially those which contain a considerable proportion of dried milk. Owing to the inclusion of fat globules, the solvent action of the ether may be almost entirely inhibited. This was notably the case with Food No. 68, which yielded only 1 per cent. of fat after twenty-four hours of ether extraction in a Soxhlet apparatus, but gave 8·5 per cent. by the Werner-Schmidt and Röse-Gottlieb methods. Even the convenient and accurate Röse-Gottlieb method fails with these refractory dried milks unless special precautions are taken—such as preliminary warming of the material with the ammonia before the addition of the alcohol, petroleum, and sulphuric ethers, etc. (McLellan, ANALYST, 1908, **33**, 353; Richmond, ANALYST, 1908, **33**, 389). In any case where doubt existed the Werner-Schmidt method was used as a confirmatory test.

The term "cellulose" is used in this report to designate carbohydrate matter not starch, insoluble in boiling water and not attacked by diastase. None of the foods examined contained a measurable quantity of crude fibre—that is to say, material remaining unaltered when boiled with 1·5 per cent. of sulphuric acid followed by 1·5 per cent. caustic soda—but all of them contained substances, probably hemicelluloses and pentosans, which resisted boiling and subsequent prolonged treatment with diastase. As it was considered important to obtain a measure of any material present in the foods which would resist natural digestion, the following method was adopted: 5 grms. of the material (extracted with ether when more than 1 per cent. of fat was present) were stirred into a thin paste with cold water and about 200 c.c. of distilled water added, after which it was brought to the boil and the boiling continued for about half an hour. The solution was then cooled to 80° C., and 5 c.c. of the malt extract (prepared by extracting a diastatic malt with three times its weight of cold water for an hour and then filtering bright) were added, and at the expiration of five minutes again brought to the boil, then cooled to 60° C. and a further 25 c.c. of the cold malt extract added. The digestion was maintained at this temperature for three to four hours and the contents of the beaker then passed through a dried tared filter-paper. The residue was repeatedly washed with water at 60° C., and when quite free from all reducing sugar with alcohol and ether, dried for several hours in the boiling-water oven and weighed in a stoppered bottle. Filter-paper and residue are then transferred to a Kjeldahl flask and the protein determined in the usual manner. This procedure is carried out in duplicate, the ash being determined on the second residue instead of the nitrogen. The first weight less the protein and ash is the figure returned as "cellulose." Most of the coagulated protein left on the filter is derived from the malt extract used to effect the conversion of the starch.

Mr. Baker points out that possibly results more nearly approaching what occurs during digestion of the food by the infant might have been obtained by subjecting the food (made up according to directions) to the progressive action of salivary diastase (ptyalin), pepsin in 0·5 per cent. hydrochloric acid solution, and pancreatic extract (trypsin, pancreatic amylase, etc.), but is of opinion that this lengthy procedure is hardly necessary considering the small quantity of "cellulose" present in the foods. With regard to the nature of the diastase present in these preparations, it should be borne in mind that the Lintner method of measuring diastase, as modified by the Institute of Brewing (*J. Inst. Brewing*, 1906, **12**, 6), records only the saccharifying power. Material which has been malted possesses in addition the property of liquefying starch-paste. Liquefying diastase is far more resistant to the action of wet or dry heat than saccharifying diastase; in fact it is possible for some of the foods—*e.g.*, Nos. 20, 26, 44, and 73—to show only slight evidence of saccharifying diastase whilst liquefying diastase is present.

Since no reliable method of quantitatively measuring this property is available, the results are recorded in qualitative terms. For the test, the change in viscosity in ten minutes at 20° C. produced by the action of 10 c.c. of a 5 per cent. cold aqueous extract of the food was noted.

In the concluding pages of Mr. Baker's Report the analytical results are discussed, and the classification of the foods according to their composition is fully dealt with. Owing to the presence in many cases of saccharifying and liquefying diastase, it was clearly a necessary feature of this inquiry to examine carefully the samples when made up according to directions. Thus a few of the foods—*e.g.*, Nos. 56 and 83—when analysed in the dry state contain large quantities of starch, but this undesirable constituent is altered for the most part to a mixture of easily assimilable maltose and malto-dextrins during the process of preparation. To condemn such a food on its analysis before being prepared for use would be unjustifiable.

The greater number of the foods examined contained only 2 per cent. of fat or less. Such foods, when prepared according to the directions given, will contain only a little more fat than that which is present in the added mixture of milk and water. Food No. 36, which contains 0·5 per cent. of fat, and is directed to be made up practically with water only, is a particularly bad instance in this respect.

Whatever the value of the mechanical part played by these foods in preventing clotting of the milk-casein, their use involves the taking of a considerable quantity of gelatinised starch. Enzymes capable of hydrolysing starch occur in the saliva, and in the pancreatic secretion (of infants). The chemical properties of the cleavage products of starch by salivary and pancreatic diastase have not been investigated to any great extent, and there is the possibility that in the case of infants there may be formed dextrins, such as α-amylodextrin, resistant to further diastatic action, and closely approaching starch in molecular weight (J. L. Baker, *J. Chem. Soc.*, 1902, **77**, 1177).

The accompanying tables give the results of analyses of the twenty-nine foods examined.

SUMMARY OF ANALYSES OF PROPRIETARY FOODS DEALT WITH IN THE REPORT.

	1.	2.	3.	4.	7.	9.	13.	14.	16.	17.	20.	21.	26.	28.	29.
Starch, per cent. ...	66·0	Trace	52·6	74·6	62·2	68·2	75·2	72·2	60·2	71·5	69·8	65·3	69·5	73·9	68·9
Hydrolysed starch products, per cent.	nil	79·7	nil	nil	nil	nil	nil	nil	nil	nil	nil	nil	nil	nil	nil
Reducing sugar as dextrose (other than lactose) per cent.	0·3	—	3·6	1·6	0·4	1·1	0·7	Trace	1·4	0·3	1·3	3·3	1·2	Trace	1·1
Lactose, per cent. ...	nil	nil	nil	nil	nil	nil	nil	nil	1·8	nil	nil	4·2	nil	nil	nil
Cane-sugar, per cent.	9·5	2·2	12·5	1·2	0·9	1·7	1·6	1·3	9·9	1·1	4·9	1·0	4·3	1·4	1·6
Fat, per cent. ...	1·0	0·1	0·8	1·0	8·2	2·2	0·1	1·0	1·6	1·3	1·5	1·6	1·6	1·1	1·5
Proteins, per cent. Nitrogen × 6·25	8·8	6·3	14·4	10·0	11·4	9·6	1·8	8·1	10·7	10·2	11·4	12·3	11·6	9·2	12·7
Water, per cent. ...	10·1	4·9	5·6	9·6	7·0	8·7	12·4	13·1	8·1	10·6	6·1	5·8	6·6	10·0	10·4
Mineral matter, per cent.	0·6	1·7	1·7	0·5	1·5	1·1 5·2	1·9 4·3	0·6 1·9	1·3 2·3	0·6 1·7	0·7 3·0	1·1 2·4	0·6 3·2	1·4 2·9	0·5 3·0
Cellulose, per cent.	1·4	3·0	4·0	1·0	6·7	8·0	Nil	21·0	14·0	1·0	1·0	7·0	Trace	6·0	13·0
Saccharifying diastase, degrees Lintner	17·0	5·0	nil	nil	nil										
Liquefying diastase	nil	+	nil	nil	nil	nil	nil	+	+	nil	+	nil	+	nil	nil
Nature of starch ...	Wheat	Nil	Wheat	Wheat	Oat	Wheat and oat	Colocasia	Arrowroot and a cereal	Wheat	Wheat	Wheat	Wheat	Wheat	Wheat	Wheat
Difference per cent. between matter soluble in water at 15° and 35° C.	2·7	—	2·3	24·3	2·0	2·3	3·6	3·6	2·1	1·0	1·7	nil	2·4	nil	nil

Summary of Analyses of Proprietary Foods dealt with in the Report—*Continued.*

	31.	32.	33.	36.	44.	51.	54.	56.	58.	60.	63.	68.	73.	83.
Starch, per cent. ...	63·6	65·6	73·6	20·0	62·6	71·1	16·6	71·4	53·4	73·3	64·9	nil	11·7	73·7
Hydrolysed starch products, per cent.	nil	nil	nil	45·3	nil	nil	21·4	nil	nil	nil	nil	53·4	10·0	nil
Reducing sugar as dextrose (other than lactose) per cent.	0·6	2·6	0·8	—	1·5	0·6	—	3·3	0·7	0·5	5·0	—	—	1·2
Lactose, per cent. ...	nil	nil	nil	nil	nil	nil	7·0	nil	nil	nil	nil	10·0	20·0	nil
Cane-sugar, per cent.	0·6	10·7	1·5	9·2	9 8	1·7	16·0	2·1	4·2	1·7	4·7	3·7	11·7	1·2
Fat, per cent. ...	7·8	1·0	1·3	0·5	1·5	2·0	6·5	1·5	1·2	1·3	2·0	8·5	16·6	0·9
Proteins, per cent. Nitrogen × 6·25	11·4	9·8	11·0	8·8	9·8	13·0	15 3	12·6	24·2	8·9	10·7	14·0	15·0	11·6
Water, per cent. ...	10·3	6 0	7·9	7·2	9·8	6·2	5·9	7·6	10·3	10·7	8·6	3·2	5·2	8·5
Mineral matter, per cent.	1·0	0·7	0·8	1·9	0·6	0·7	4·0	0·8	2·1	0·7	1·5	4·1	3·8	1·1
Cellulose, per cent.	3·0	2·1	2·4	3·1	3·0	3·6	4 6	1·6	3·4	1·8	2·4	nil	2·5	0·9
Saccharifying diastase, degrees Lintner	nil	7·0	7·0	2·0	Under 1	nil	nil	5·0	6·0	Under 1	2·0	nil	Under 1	18·0
Liquefying diastase	nil	+	+ Sl	+	+	nil	nil	Strong	+ Sl.	nil	Strong	nil	Strong	Strong
Nature of starch ...	Oat	Wheat	Wheat	Wheat much distorted	Wheat	Wheat and oat	Probably wheat much altered	Wheat	Bean (lentil)	Wheat	Wheat and banana	—	Wheat	Wheat
Difference per cent. between matter soluble in water at 15° and 35° C.	nil	4·1	1·6	—	3·4	2·2	—	13·0	2·2	nil	3·5	—	3·4	—

G. W. M-W.

REVIEWS.

THE MICROSCOPY OF DRINKING-WATER. By GEORGE CHANDLER WHIPPLE. With a Chapter on the Use of the Microscope, by John W. M. Bunker, Ph.D. Third Edition, rewritten and enlarged, with coloured plates. New York: John Wiley and Sons. London: Chapman and Hall, Ltd. 1914. Price 17s. net.

The fact that after a period of three years from the time of the second edition of this work a third edition, expanded and rewritten, should be called for, is sufficient evidence of its wide utility.

The further investigation is carried into questions of water-supply and the kindred subject of stream pollution, the more it becomes evident that for their proper understanding a correlation of biological, with chemical and physical data, is required.

The biological factors have been systematically studied from this point of view only during comparatively recent years, and Professor Whipple's book is one of a small number in the English language which deals with the micro-organisms other than bacteria, and with their practical significance.

The book is divided into two parts, the first comprising methods of examination and the broad results of study of lakes and reservoirs, the second part being systematically descriptive of the chief genera and species of micro-organisms met with in water-supplies. The chapters on the collection and examination of samples will be most useful to the student and beginner.

The subject of Limnology, or the study of lakes and such-like bodies of water, is one to which more attention might profitably be paid in this country. Sundry obscure troubles, which sometimes arise in water taken apparently from a satisfactory source, might then be better understood and dealt with.

In Chapters VII. to XII. the important questions are discussed of the relation of micro-organisms in lakes to the dissolved gases present, the temperature, the season, etc. The question of dissolved gases is one of great importance, and is, of course, conditioned itself by many chemical and physical factors. The process of photosynthesis as affected by algæ and aquatic plants here plays a large part.

Chapter XII., on odours in water-supplies, is of great practical importance, and an original attempt is made to obtain quantitative estimates of odours and offensiveness. Some paragraphs in relation to this subject are reprinted from the author's well-known work on "The Value of Pure Water."

The opening paragraphs of Chapter XIII., on the storage of surface-water, certainly deserves the emphasis given it by italics—*i.e.*, "To obtain a permanently safe and satisfactory surface-water supply without filtration, the rainfall must be collected quickly from a clean watershed and stored in a clean reservoir."

The conclusions reached in the subsequent chapter, on soil-stripping, would seem to be at variance with the spirit of this statement, in that it is shown that in many cases money is better spent in filtration than in such a preparation of the reservoir.

The second part of the book must be criticised, if at all, after practical use.

The general descriptions appear adequate, and copious references are given, and a useful glossary for those readers who are not trained biologists.

A word, finally, must be said about the illustrations. The actual photo-micrographs in the text cannot be regarded as successful. The plates at the end are an improvement upon those in the second edition by the introduction of colour. There is, however, a certain lack of definiteness and detail in outline and structure in many cases which is disappointing. The production of plates is expensive, but it might perhaps be suggested that in a new edition the second part of the book should be made into a separate volume, and the plates further revised and improved even at some additional cost. The micro-organisms are things of beauty, and the steel engraving of old-fashioned works gave a better idea than the somewhat blurred impressions at the end of the book under review.

Many of the statements in the chapter on the use of the microscope (contributed by John W. M. Bunker) require revision and reconsideration—*e.g.*, particularly in regard to illumination, where the author is at variance with many established authorities.

Taken as a whole, however, the book is well worth careful study and continuous reference to by all those engineers, biochemists, and health officers who are interested in obtaining and conserving a satisfactory water-supply for the modern community.

G. J. Fowler.

Clean Water and How to Get It. By Allen Hazen. Second edition. New York: John Wiley and Sons. London: Chapman and Hall, Ltd. 1914. Pp. 192. Price 6s. 6d. net.

This work deals with the supply of pure drinking-water to towns, as practised in America. It is not written for the expert, but for the ordinary individual, to help him to understand the objects to be accomplished and the means of carrying them out.

The chief subjects dealt with are the raw supplies, purification and sterilisation, engineering data for filters and pressure of supply, measurement and waste, and financial aspects. The subject of the chemical and bacteriological quality of the purified water is not included. The matters which must be removed in order to render a supply clean, the author states to be—taste, odour, colour, turbidity, hardness, iron, sewage pollution. The description of the filtration processes serves to bring out a great difference between American and English practice, in that a list of fifty-three large and small towns is given where mechanical filters are in use, while only seventeen places use slow sand filters. This difference is further emphasised by the statement that the greater part of the water supplied to towns is treated by some process of disinfection. For this latter purpose bleaching powder is the substance most extensively used. The author recommends that disinfection should take place after filtration, and that before filtration a coagulant, such as aluminium sulphate, should be used, allowing time for settlement.

The author is well known as having taken part in the experiments of the Massachusetts State Board of Health, and therefore it might be expected that he would take the view that all sewage should be purified before being turned into a drinking-water stream. But no! It is stated that the keeping of all sewage out of rivers is not a practical proposition and is not necessary, in fact not even desirable, and that when the discharge of crude sewage into a river does not produce a local nuisance, there is no justification for its purification just because a waterworks intake is situated lower down. Fortunately this is not the most general view in this country, otherwise some of our water authorities would find their task even more onerous than it is now.

The first edition of this book was published seven years ago, and in this, the second edition, chapters have been inserted on disinfection, already noticed, and on "red-water." The latter is the name given to a water when highly charged with oxide of iron, arising from a soft water acting on iron mains and service pipes, and corroding them or forming "tubercles." A general preventative against this action is the addition of quicklime to the water.

Altogether the beginner will find the book very readable, and he should obtain a clear idea of all the factors that may have been required to produce the tap-water he uses. The book is provided with many illustrations, mainly of engineering features, and a somewhat incomplete index.

J. H. JOHNSTON.

RECUEIL DE LÉGISLATIONS BEURRIÈRES ET MARGARINIÈRES CONCERNANT LA RÉPRESSION DES FRAUDES DANS LE COMMERCE DU BEURRE. 1re partie. Par Dr. A. J. SWAVING. La Haye: W. P. van Stockum et Fils. 1914. 3.50 f.

This work, published under the auspices of the permanent Bureau of the Fédération Internationale de Laiterie, contains a full collection of the legislation of fifteen European countries with regard to the manufacture and sale of butter and margarine. The work has been so arranged that the development of such legislation in each country can be closely followed, and the separate sections are brought to a conclusion by a table, in which the existing conditions are set out in an easily comparable manner.

Dr. Swaving appears to have carried out his work in a most careful and painstaking manner, and there is no unnecessary matter.

It is to be recommended to all those who are interested in the control of butter and margarine arriving on the English market. Its general value is enhanced by the fact that the languages have been confined to English, French, and German.

C. REVIS.

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THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

NOTES ON THE DETERMINATION OF THE CINEOL CONTENT OF EUCALYPTUS OILS.

By HENRY G. A. HARDING.

In Schimmel's semi-annual report (1908) some notes are recorded on the resorcinol method for the determination of cineol. Every worker with this method soon realises its unreliability when used on the virgin or ordinary rectified oils, and it is no uncommon thing to meet with samples of oil, containing no cineol, which nevertheless give quite considerable readings. Recently I had occasion to work with a pure white E. Dives oil, free from cineol, which showed, however, 32 per cent. by using the resorcinol test.

The rectification suggested by Schimmel—viz., using for the test only that portion of the oil distilling between 170° to 190° C.—seems to remove this difficulty. Notwithstanding this, the results are always slightly high. This is perhaps negligible when the percentage does not exceed 50; for higher percentages I have found that the difference between the apparent and true percentages warrant some suggestions for overcoming the difficulty, which seems to be due to some retention by the resorcinol solution.

By using a warm 55 per cent. solution of resorcinol instead of a 50 per cent., better results are obtained. It is well to remember that in very cold weather solutions of over 50 per cent. strength are apt to crystallise, and also is always the difficulty that with high percentages of cineol (above 75 per cent.), small flakes of the euco-resorcinol compound separate, and, rising to the surface of the concentrated resorcinol solution, lie at the junction of the two liquids, and prevent an accurate reading.

My experiments show that this method is most reliable when the percentage of cineol does not exceed 40 to 50 per cent. High percentage oils require to be diluted, and for this purpose Schimmel recommends turpentine. Unless great care is taken, the use of turpentine is likely to produce serious errors. Various samples of different qualities were tried, and all were found to contain substances absorbed by resorcinol.

After many trials I found that by using as a diluent the fraction of turpentine distilling between 156° to 160° C., the objection was overcome. To confirm this, the first fractions (below 150° C.) were collected and tested with resorcinol, and also the higher fractions (over 160° C.), as well as the residual oil in the distillation flask, and all showed appreciable though varying absorptions, according to the variety of turpentine used. Of these, the first distillate showed least absorption, the higher distillate more, and the residuum by far the greatest, the absorption obviously being due to oxidation products of the terpenes. It is convenient to distil 200 c.c. of ordinary turpentine in a 250 c.c. round-bottom distillation flask, fitted with side-tube, by means of direct flame. The fraction distilling between 156° to 160° C. may be kept indefinitely.

One hundred c.c. of the oil to be tested for cineol are distilled in a flask of 150 c.c. capacity, reserving the distillate collected from 170° to 190° C., and then diluting to 100 c.c. with the distilled turpentine. If a trial shows the percentage of cineol to be above 70 per cent., the distilled cineol is diluted further with the turpentine, so that the percentage is not over 50. The temperature is noted, and 6 to 10 c.c. shaken with warm 55 per cent. resorcinol solution. After five minutes' shaking more solution is added, so as to bring the oil to the graduated neck. It is then cooled, and the volume read.

* * * * *

THE ESTIMATION OF CANE-SUGAR AND LACTOSE IN CONDENSED MILK BY MEANS OF ACID MERCURIC NITRATE.

By CECIL REVIS and GEORGE A. PAYNE.

The use of acid mercuric nitrate, both as a clarifying and also as an inverting agent in the analysis of condensed milk, is due originally to Harrison (Analyst, 1904, **29**, 248) and the method has since been rediscovered by Cochran (*J. Amer. Chem. Soc.*, 1907, **29**, 555). Both these workers give similar inversion factors (142·66 Harrison, and 142·68 Cochran) in the Clerget formula. During the course of an investigation we hah reason to suspect that this factor was not quite correct, and a careful examination of the method has confirmed this.

The apparent error in Harrison's factor is not due to any fault in experimental procedure, as his work was most carefully carried out, but is due to the fact that he obtained his factor under conditions which are not those of the actual analysis of condensed milk.

In Harrison's experiments a direct reading of the sugar solution was made and the acid mercuric nitrate then added, inversion carried out, and the invert reading obtained.

Now, in practice, the mercuric nitrate is necessarily added before the direct reading is made, and an interval of at least ten minutes must elapse before the direct reading is made. Cochran states that as long as the sugar solution is kept below 10° C., no inversion of the cane-sugar takes place for several hours; but this, in our opinion, is not correct, and the inversion commences immediately and is probably largely the cause of the different factor at which we have arrived. It must also be remembered, that, as Richmond has shown, the precipitation of protein by acid mercuric nitrate is not complete, and the residual protein affects the polarimeter readings.

The following figures show the effect of keeping a solution of cane-sugar at 0° C. (melting ice) for several hours:

Sugar Solution.	Water.	Acid Mercuric Nitrate.	Reading (Ventzke).	
			At once.	Five hours later.
C.C.	C.C.	C.C.		
50	7·5	—	43·77	—
50	5·0	2·5	43·75	42·80
50	2·5	5·0	43·70	41·34
50	—	7·5	43·43	40·93

It will be seen that not only is there distinct inversion at 0° C. in five hours, but that with increasing quantities of acid mercuric nitrate there is an immediate effect as well. There also appeared to be a slight raising of the reading in the case of lactose when acid mercuric nitrate is added.

In order to obtain a factor which would be free from these sources of error, some very careful experiments have been carried out both with solutions of pure cane-sugar and with an artificial condensed milk made by adding pure cane-sugar to dried separated milk. The range of concentration of the sugar was kept in the neghbourhood of that which would occur in dealing with ordinary condensed milk.

The great ease and utility of the acid mercuric nitrate method is our excuse for this investigation. In making these experiments, every source of error has been eliminated as far as possible, the polarimeter used being carefully checked by means of quartz plates, kindly lent to us by Mr. A. Ling and Mr. Eynon.

There is a source of error, which may occur in this method of estimating the sugars, which is due to the occlusion of sugar in the precipitated curd, and unless care be exercised, the error from this source may be large.

In order to avoid the use of special measuring vessels graduated at unusual temperatures, and which are not likely to be found in the ordinary laboratory, we have adopted the following procedure in order to avoid such difficulty:

65·085 grms. of the condensed milk are weighed out and washed into a graduated flask containing 250 true c.c. adjusted at 15° C. (the ordinary standard Charlottenburg flask), with sufficient water to bring the volume to about 220 c.c. This is then heated in boiling water for five to ten minutes, or else allowed to stand overnight. In either case the volume is eventually made up to the mark at 15° C. After well mixing, 100 c.c. are measured out into a long stoppered cylinder with a 50 c.c. pipette (50 c.c. = 50 grms. of water at 15° C.), after which 10 c.c. (true c.c. at 15° C.) of acid mercuric nitrate are added, and the whole violently shaken for thirty seconds till the curd is in the finest possible state of division. The cylinder is allowed to stand for five minutes at 15° C., and the contents then filtered through a dry filter. As soon as sufficient for the direct reading is through, its temperature is raised (if necessary) to 20° C., and the polarimeter-tube filled and the reading taken at that temperature. Twenty-five c.c. of the remainder of the filtrate are placed in a 50 c.c. stoppered flask, counterpoised, and then heated in boiling water for eight minutes, cooled, readjusted to the original weight, filtered if necessary, and the reading taken as near 20° C. as possible.

The readings are corrected for the increase in volume due to the added mercuric nitrate, less the volume of the fat and protein precipitated.

This correction (C) will be:

$$C = 10 - \left(\frac{(F \times 1{\cdot}11) + (P \times 0{\cdot}82)}{100} \times 26{\cdot}034\right) \text{c.c.}$$

and the readings must be multiplied by $(100 + C)$ to give the correct results for the normal weight. The following formulæ then give the percentage of sugars:

$$\text{Cane-sugar per cent.} = \frac{(D - I)\ 100}{(R_c - \frac{T}{2})} = S,$$

$$\text{and Lactose per cent.} = (D - S) \times \frac{R_L}{26{\cdot}034} \times 100,$$

where D = corrected direct reading,
I = corrected invert reading,
$\left.\begin{matrix} R_c \\ R_L \end{matrix}\right\}$ = the inversion and lactose factors,
and T = temperature of invert reading.

It is assumed that a Schmidt and Haensch quartz-wedge compensating polarimeter is used, reading in cane-sugar units, and so graduated that if 26·048 grms. of cane-sugar are dissolved in 100 Möhr c.c. at 17·5° C., and polarised at 17·5° C., the reading will be 100 scale divisions. The change in the above formulæ of 26·048 to 26·034 is necessitated by the analytical procedure adopted.

As the result of many very careful experiments, we have obtained the following values for R_c and R_L at sugar concentrations which will be realised in dealing with condensed milk:

$$R_c = 141{\cdot}71$$
$$R_L = 0{\cdot}3086$$

In order to check the correctness of these factors, a similar series of experiments was made, using as a precipitant a phospho-tungstic acid mixture suggested to us by Mr. Macara, in which no inversion of the cane-sugar occurs, and inversion is carried out by the ordinary Herzfeld process on the clear filtrate.

This series gave the following results:

$$R_c = 142{\cdot}55$$
$$R_L = 0{\cdot}3165$$

These figures approximate closely to those found by other observers, and establish the correctness of the new factors.

We have carried out a parallel series of experiments on condensed milk alone, and on the same milk with additions of invert sugar or glucose syrup, in order to see if the figures agreed when using acid mercuric nitrate and the phospho-tungstic acid precipitant. The invert sugar used was specially prepared, and contained 82 per cent. of invert sugar and only a trace of residual cane-sugar.

The results were as follows:

Sample.	Acid Mercuric Nitrate.		Phospho-Tungstic Acid Precipitant.	
	Cane-Sugar.	Apparent Lactose.	Cane-Sugar.	Apparent Lactose.
Condensed milk	38·69	13·78	38·81	13·70
„ 99 parts + I.S. 1 part ...	38·48	12·96	38·37	13·41
„ 98 „ + I.S. 2 parts ...	38·26	12·62	38·34	12·87
„ 97 „ + I.S. 3 „ ...	37·74	12·34	37·85	12·57
„ 99 „ + G[illegible] 1 part ...	38·63	15·24	38·50	15·63
„ 98 „ + G[illegible] 2 parts ...	38·30	17·04	38·26	17·21

The agreement between these two series is quite close. The percentage of apparent lactose discloses quite easily the presence of glucose syrup, as in any well-made condensed milk the true percentage of lactose can be calculated with a fair degree of accuracy as being $\frac{13}{24}$ths of the non-fatty milk solids present.

If the lactose be also determined by Fehling's method, the presence of invert sugar is similarly indicated, and by the usual equations the relative percentages of invert sugar and lactose could be calculated with sufficient accuracy.

The figures obtained for the apparent lactose by Fehling's method in the above series were:

13·59 per cent.	15·62 per cent.	14·72 per cent.
14·51 „	16·48 „	15·10 „

It can be seen that by combining the polarimetric method with a copper determination of the apparent lactose, evidence as to presence of glucose syrup or invert sugar caused by careless condensation, is easily obtainable.

The acid mercuric nitrate is made as follows: pure mercuric oxide is dissolved in twice its weight of nitric acid (sp. gr. 1·42), and the resulting solution diluted to five times its volume with water.

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Alcohol Estimation in the Tropics. K. C. Browning and C. T. Symons. (*J. Soc. Chem. Ind.*, 1914, **33**, 819-821.)—It has long been apparent that the usual brass Sykes' hydrometer does not give correct indications of the strength of aqueous alcohol at tropical temperatures (80°-90° F.). In India a set of tables calculated by Bedford for use with glass Sykes' hydrometers has been adopted. The authors have carried out a series of comparative investigations with the metal and glass types of Sykes' instrument on a range of alcoholic solutions carefully standardised by means of pyknometer estimations. Wade and Merriman's pyknometer (*J. Chem. Soc.*, 1912, **101**, 2429) was found to be by far the most satisfactory, and every precaution was taken to insure accuracy in the temperature adjustments. The results for alcoholic strength were calculated from the specific gravities at 60° F. by Hehner's tables; the hydrometer readings were taken at 84°-88° F. The aqueous alcohols studied ranged from 1·06 to 88·96 per cent. by weight. It was established that the brass hydrometer, when used with the tables supplied by the makers of the instrument, was quite inaccurate, particularly with the more dilute spirits, and was not even approximately satisfactory at any strength of alcohol. Bedford's tables used with glass hydrometers gave more satisfactory results except for the more dilute alcohols. A further series of pyknometer estimations was conducted at 35° C., using the new United States tables. The authors express the opinion that these tables are among the most accurate that have been published, and that all work on alcohol should be based on them. Compared with the values calculated by the United States tables, the brass hydrometer employed with the ordinary tables is also useless, but when used with Bedford's tables it gives very fair results indeed. Hydrometers should be graduated to show the percentage of alcohol either by weight or volume, and the concept of proof spirit should be discarded. The Zeiss immersion refractometer affords a very convenient means for the rapid estimation of alcoholic strengths up to about 35 per cent.; above that strength its accuracy is not satisfactory. The authors have constructed a table for use with the refractometer at 30° C. based on pyknometric estimations at 35° C. calculated by the United States tables. It is noted that the glass hydrometers undergo somewhat large and irregular variations in course of time. Hydrometers constructed for use in the tropics should be standardised at 30° C., instead of at the usual temperature of 51° F. J. F. B.

Estimation of Antipyrin. W. O. Emery and S. Palkin. (*J. Ind. and Eng. Chem.*, 1914, **6**, 751-752.)—I. *Antipyrin alone or free from substances yielding an oxide capable of extraction with chloroform:* The weighed quantity of the sample (not exceeding 0·25 grm. of antipyrin) is mixed with 20 c.c. of water, 5 c.c. of alcohol-free chloroform, and 15 to 20 c.c. (or a slight excess) of $\frac{N}{5}$ iodine solution, and the mixture vigorously shaken at intervals for five minutes. The free iodine is then removed by adding a few drops of thiosulphate solution, and the iodo-antipyrin extracted with three successive portions (25 c.c. each) of chloroform. The extracts are separately washed with 5 c.c. of water, filtered, and evaporated together, and the residue dried for 30 minutes at 110° C. The weight of iodo-antipyrin multiplied by the factor 0·5992 gives the amount of antipyrin. II. *Antipyrin in admixture with acetanilide, phenacetin, sulphonal, or other substances incapable of yielding an iodine derivative insoluble in dilute acids:* The aqueous solution of the sample (not exceeding 0·25 grm. of antipyrin) in about 50 c.c. of water is shaken with 20 c.c. of strong hydrochloric acid and 50 to 60 c.c. of $\frac{N}{10}$ iodine solution, and the mixture allowed to stand for at least three hours. The clear supernatant liquid is decanted through an asbestos filter, and the tarry periodide precipitate washed by decantation with eight or nine portions of 20 c.c. each of 5 per cent. hydrochloric acid. It is then dissolved in 20 c.c. of warm methyl alcohol (free from ethyl alcohol and acetone), the solution transferred to a separating funnel, and the flask rinsed with more of the same solvent, the washings being passed through the filter. The solution is treated with 5 c.c. of sodium bicarbonate solution (which converts the di-iodo-antipyrin into iodo-antipyrin, $C_{11}H_{11}N_2IO$) and 50 c.c. of water, and the funnel shaken vigorously at intervals for five minutes, after which the excess of iodine is removed, the iodo-antipyrin extracted with three portions of 40 c.c. of chloroform, the extracts each washed with 10 c.c. of water, passed through a dry filter, and evaporated in a tared flask. The method is not applicable to mixtures containing caffeine or pyramidon. C. A. M.

New Reaction of Cocaine. F. Pisani. (*Rend. Soc. Chim. Ital.*, 1914, **6**, 132; *Annili Chim. Appl.*, 1914, **2**, 51; through *J. Soc. Chem. Ind.*, 1914, **33**, 982.)—The free alkaloid or its hydrochloride, when heated with a few drops of concentrated sulphuric acid containing 2 per cent. of hexamethylenetetramine, gives a wine-red coloration, becoming more intense as the temperature rises; after a short time the colour disappears and a brownish-grey sediment is left. The reaction, which is due to benzoic acid, and is not given by atropine, quinine, cinchonine, brucine, strychnine, morphine, apomorphine, codeine, or narcotine, is distinct with 0·001 grm. of cocaine. Papaverine under similar conditions may also give a wine-red coloration, changing, however, after a short time, to yellow, reddish-brown, and orange. In doubtful cases papaverine may be identified by the violet coloration it gives with sulphuric acid alone, a reaction not given by cocaine.

Earth-nut Butter. C. A. A. Utt. (*J. Ind. and Eng. Chem.*, 1914, **6**, 746-747.)—Earth-nut or pea-nut butter is prepared from the slightly roasted kernels of the nuts. Twenty-three commercial samples contained from 44·74 to 53·64 per cent. of oil, with an $[n]_{D\ 25^\circ C.}$ 1·4680 to 1·4707, and iodine value of 88·04 to 94·36; whilst three samples prepared in the laboratory yielded 50·65 to 52·03 per cent. of oil, with an $[n]_{D\ 25^\circ C.}$ 1·4685 and iodine value 88·75 to 90·03. No evidence was obtained of the addition of any foreign oil to the commercial products. C. A. M.

Valuation of Feeding-Stuffs by Means of Chemical Analysis. A. Smetham. (*J. Roy. Lancashire Agric. Soc.*, 1914.)—The results of analyses of a large number of feeding-stuffs are shown in the following table (*cf.* ANALYST, 1910, **35**, 54). The analyses of a number of materials are included which have not previously appeared, and which will be found useful for purposes of reference. "Albuminoids" represent total nitrogen, multiplied by 6·25. "Digestible carbohydrates" include all non-nitrogenous organic matter, except woody fibre and fat. The "food units" are calculated on the assumption that oil and albuminoids are of equal value, and together are 2·5 times as valuable as carbohydrates. When a food has been ascertained to have poisonous properties, the fact is stated in the tables, but the absence of such a statement does not necessarily imply that the foods are wholesome:

Name.	Water.	Oil.	Albuminoids.	Digestible Carbohydrates.	Woody Fibre.	Mineral Matters.	Sand and Silica.	"Food Units."
Wheat, English:								
Garton's	12·90	2·85	12·19	69·06	1·30	1·70	—	106
Hardcastle white	13·90	2·35	12·19	68·01	1·75	1·80	—	104
Wheat, Burma	10·40	1·77	15·75	68·60	1·88	1·60	—	112
„ Danubian	9·35	2·03	15·75	69·22	2·10	1·55	—	113
„ Indian, hard	9·94	1·50	9·81	75·40	1·50	1·85	—	104
„ „ soft	13·33	1·76	10·50	69·42	2·10	2·89	—	100
Flour patents, American	12·00	1·37	12·75	73·46	0·07	0·35	—	109
„ bakers	11·90	1·30	13·62	72·63	0·10	0·45	—	110
„ seconds	11·40	4·56	17·94	58·42	4·03	3·65	0·10	115
„ thirds	11·20	3·56	18·37	61·66	2·46	2·75	0·05	116
„ standard (Rolleston)	13·90	1·77	11·88	70·70	0·80	0·95	—	105
„ brown, germ	11·40	2·97	14·25	69·51	0·67	1·20	—	113
Sharps	11·80	3·30	13·12	58·95	8·73	4·10	0·30	100
„	11·05	5·13	18·01	54·36	6·60	4·85	0·20	112
Wheat, pollards	10·70	3·60	14·25	56·73	8·57	6·15	1·55	101
„ „	11·65	5·13	17·69	55·59	5·43	4·51	0·15	113
„ „ La Plata	11·30	4·17	19·13	55·73	5·42	4·25	0·10	113
„ germ	12·01	7·70	21·41	51·18	3·90	3·80	—	124
„ „ (good)	13·30	8·63	31·37	42·00	0·60	4·10	—	142
Bran	11·45	4·33	16·46	57·01	6·50	4·25	—	109
„ flattened	12·25	4·30	15·37	58·86	5·37	3·85	0·20	107
Cockle	11·45	3·16	12·62	55·94	10·83	6·00	2·25	95
Wheat screenings	11·35	4·67	12·37	61·14	5·77	4·70	1·65	104
Ground winnowings	8·20	1·90	9·31	33·95	9·63	37·01	29·30	61
Barley grain, average of:								
English (42 analyses)	14·95	1·46	8·59	67·96	4·46	2·58	0·39	93
Azoff	12·95	2·50	13·25	64·13	4·37	2·80	1·05	103
Smyrna	12·30	2·27	11·88	63·92	5·73	3·90	1·60	99
Canadian	13·30	2·07	12·12	65·13	4·48	2·90	0·85	100
Danubian	12·40	1·93	11·00	68·00	4·22	2·45	0·50	100
Offal, from pearl-barley meal	10·10	3·90	14·37	58·81	7·67	5·15	2·40	104
Oats, crushed	13·45	6·77	11·12	56·92	9·14	2·60	0·85	102
Oatmeal	8·14	7·03	13·17	66·97	2·75	1·94	—	117

Name.	Water.	Oil.	Albuminoids.	Digestible Carbohydrates.	Woody Fibre.	Mineral Matters.	Sand and Silica.	"Food Units."
Oatmeal, Irish	7·85	9·53	14·38	64·77	1·57	1·90	—	124
Oat shudes	9·30	0·83	1·31	52·74	31·87	3·95	3·10	58
Feeding oatmeal	8·40	5·10	10·12	60·13	12·60	3·65	1·65	98
"Meal seeds" (refuse from oatmeal)	9·10	5·17	10·12	57·78	14·53	3·30	1·70	96
"Oat feed" (refuse from oatmeal)	10·05	5·90	7·63	52·49	19·83	4·10	1·90	86
Rye flour	12·15	1·77	9·75	74·67	0·16	1·50	0·15	103
Dari, Indian	10·70	3·03	7·75	74·36	2·21	1·95	0·25	101
„ seed meal	11·50	4·93	10·25	67·75	2·02	3·55	0·85	105
White Jowaree meal (Guinea corn), Karachi	10·65	3·43	9·75	71·53	1·79	2·85	0·75	104
Millet, Rangoon	12·40	3·13	10·62	70·34	1·41	2·10	0·25	104
„ Russian	12·00	4·43	10·50	61·65	8·37	3·05	1·60	99
„ Manchurian	12·80	3·83	9·38	70·58	1·71	1·70	0·05	104
American white Kaffir corn	11·90	3·30	10·63	71·44	1·48	1·25	0·05	106
Maize:								
new crop	17·45	4·06	9·25	66·14	1·50	1·60	0·05	99
old, average	11·25	4·80	10·20	70·17	1·85	1·73	—	108
Danubian	12·85	4·23	9·75	70·90	1·17	1·10	—	106
Turkish	15·95	4·63	9·12	67·30	1·65	1·35	—	101
River Plate	13·55	5·07	10·12	68·88	1·13	1·25	—	107
North American, mixed	12·55	4·37	10·62	69·71	1·50	1·25	—	107
Corn meal	12·45	4·20	10·01	70·13	1·96	1·25	—	105
Maize germ	14·65	25·47	14·88	31·00	7·45	6·55	0·25	132
„ „	12·05	8·66	15·13	55·07	5·99	3·10	0·10	114
„ „ meal	10·35	14·66	26·16	40·60	6·93	1·30	0·20	143
„ „ „	10·55	8·87	20·12	55·90	3·81	0·75	0·05	128
„ „ cake	9·50	6·86	15·07	57·91	5·26	5·40	0·15	113
„ residue (chemically treated)	11·75	18·66	29·31	20·75	6·33	13·20	8·60	141
„ tailings	12·25	10·36	10·87	57·01	6·26	3·25	0·20	110
Maizone flake	12·01	0·85	8·50	77·69	0·60	0·35	—	101
Flaked maize (brewing)	6·30	0·87	10·62	81·56	0·30	0·35	—	110
„ „ (feeding)	12·90	3·57	9·38	71·28	1·77	1·10	—	104
Maize grits	12·50	1·53	7·50	77·22	0·60	0·65	—	100
Gluten meal (by-product)	8·50	19·47	36·01	31·82	2·15	2·05	0·10	170
„ „ „	8·60	10·60	30·12	44·16	4·62	1·90	0·10	146
„ „ „	9·65	4·53	32·25	51·57	1·25	0·75	0·10	143
Hominy meal	9·80	9·46	10·88	62·76	4·05	3·05	0·10	114
„ feed	10·25	8·07	10·87	64·86	3·10	2·85	0·15	112
„ „	7·15	10·73	12·75	60·40	5·72	3·25	0·10	119
Maize bran	11·05	13·37	12·75	52·22	6·81	3·80	—	117
„ „	13·75	3·10	6·13	62·94	12·93	1·15	—	86
Corn flour	11·25	4·10	6·90	76·77	0·23	0·75	—	104
„	11·35	0·27	0·50	87·50	0·03	0·35	—	89
Germ corn flour	11·90	4·40	8·38	73·47	0·40	1·45	—	105
Corn cob meal	11·05	0·80	2·00	56·18	28·52	1·45	0·30	63

Name.	Water.	Oil.	Albuminoids.	Digestible Carbohydrates.	Woody Fibre.	Mineral Matters.	Sand and Silica.	"Food Units."
Rice (cleaned)	11·80	2·40	8·01	75·88	0·76	1·15	—	102
,, flour (from hand-picked cleaned rice)	12·65	2·20	7·75	75·34	0·66	1·40	—	100
,, husks (hand-picked)	9·70	1·16	3·25	32·44	39·90	13·55	13·20	43
,, flour from Burma rice	11·30	0·57	7·00	80·65	0·13	0·35	—	99
,, flour, Canadian ...	12·75	0·93	7·00	78·45	0·17	0·70	—	98
,, meal or bran English	9·95	10·43	13·94	48·95	8·03	8·70	4·05	110
,, ,, Rangoon	9·10	15·46	13·31	47·00	6·23	8·90	1·25	119
Rice meal or bran:								
Chittagong	8·99	15·86	13·00	41·98	7·53	12·64	5·09	114
Saigon	8·70	11·40	11·62	59·46	3·27	5·55	1·50	107
Madras	8·20	15·07	11·50	45·60	8·33	11·30	6·20	112
Italian	7·70	8·21	8·86	41·13	22·20	11·90	8·15	84
Canadian	10·15	9·73	10·62	46·00	14·80	8·70	4·10	97
Australian	9·15	17·83	13·00	44·29	5·83	9·90	2·75	121
Siam	9·25	16·60	11·75	50·32	3·83	8·25	1·55	121
Demerara	9·00	21·37	12·63	41·35	5·80	9·85	4·65	126
Rice meal (chiefly ground shudes)	9·55	1·93	2·87	36·15	30·20	19·30	17·90	48
,, ,, Polish... ...	9·50	10·07	11·62	63·90	0·61	4·30	0·25	118
Beans: English	14·14	1·86	28·12	46·70	6·14	3·04	—	121
Chinese, split	13·55	2·17	25·75	48·20	6·88	3·45	0·30	118
Large Japanese haricot (old crop)	14·25	1·80	20·25	56·53	3·67	3·50	—	111
Pai-Yin, Burma (Vigna Catiang)	10·35	1·07	23·00	56·83	5·05	3·70	0·05	117
White African (Vigna Catiang)	14·85	1·67	24·75	52·80	2·78	3·15	0·05	119
Rangoon red (sometimes poisonous)	11·75	1·37	20·00	59·16	4·02	3·70	0·10	113
Soya: Manchurian ...	11·80	17·60	38·13	22·06	5·26	5·15	0·55	161
Korean	10·35	17·30	38·13	22·89	6·93	4·40	—	161
Caucasus	10·25	19·17	34·63	24·27	6·68	5·00	0·10	158
Egyptian	10·05	19·33	35·88	22·72	5·72	6·30	0·15	161
Argentine (from Manchurian seed)	14·05	19·90	32·13	24·34	4·08	5·50	0·05	154
China Soya bean cake ...	17·00	9·00	40·50	23·78	4·47	5·25	0·40	147
,, ,, ,, ...	17·75	5·00	47·50	18·46	5·49	5·80	0·35	150
English ,, meal...	3·85	11·33	43·25	30·77	5·45	5·35	0·25	167
,, ,, cake...	12·70	11·07	38·82	26·51	5·85	5·05	0·35	151
,, ,, meal (extracted)	11·82	1·50	44·00	32·08	5·38	5·22	0·44	146
Chinese bean cake (rolled)	8·65	17·60	39·00	25·48	4·42	4·85	0·15	167
,, ,, (crushed)	9·10	10·23	42·75	27·82	4·90	5·20	0·20	160
,, ,, (from Southern ports)	11·65	5·60	46·12	25·46	4·47	6·70	1·20	155
Manchurian bean cake ...	15·60	8·60	41·37	24·81	4·37	5·25	0·20	150
Peas: English	14·20	1·71	22·50	52·59	5·99	3·01	—	113

Name.	Water.	Oil.	Albuminoids.	Digestible Carbohydrates.	Woody Fibre.	Mineral Matters.	Sand and Silica.	"Food Units."
Peas: Canadian	13·95	1·93	24·31	52·91	4·20	2·70	—	119
Ground green Indian ...	12·15	2·23	27·06	48·05	7·66	2·85	—	121
Calcutta, green	14·50	1·80	25·75	47·94	7·01	3·00	0·25	117
Chinese ,, ...	12·05	1·13	22·50	54·68	6·89	2·75	0·35	114
Russian	17·45	1·83	23·62	48·57	5·53	3·00	0·55	112
Pea meal	13·70	1·86	24·87	52·97	3·90	2·70	0·05	120
,, ,, (refuse from split peas)	9·25	3·13	20·37	47·59	15·91	3·75	0·45	106
,, hulls, white	12·20	0·73	5·25	31·05	47·92	2·85	0·10	46
,, ,, (from Calcutta white peas)	8·45	0·50	6·63	35·16	46·41	2·85	0·05	51
Horse gram, Kulthi, Kooltee or Kult peas (*Dolichos biflorus*)	11·05	1·10	23·37	54·27	5·11	5·10	1·50	115
Bengal horse gram (*Cicer arietinum*), whole seed	9·30	4·67	21·25	53·66	8·11	3·01	0 05	118
Bengal horse gram (split)	12·95	5·43	21·88	55·71	1·13	2·90	0·15	124
Indian "peas" — mutter (*Lathyrus Sativus*) (poisonous)	13·10	1·46	31·62	42·54	8·13	3·15	—	—
"Winter tares" from Smyrna (probably *Lathyrus Sativus*)	10·55	1·46	22·12	58·49	4·08	3·30	0·50	—
Tares, Russian	12·90	1·70	23·26	50·67	6·77	4·70	1·30	113
"Bombay tares"—pigeon pea (*Cajanus Indicus*)	12·00	2·07	20·38	55·67	6·38	3·50	0·05	112
Lentils, average	9·80	2·01	25·50	59·03	1·01	2·65	0·05	127
,, Syrian	10·55	1·90	24·25	56·53	3·87	2·90	0·20	122
Locust beans	13·40	1·01	4·87	72·24	5·68	2·80	0·05	87
,, ,, Algerian ...	11·85	1·03	5·94	71·43	7·10	2·65	—	89
,, bean meal	12·05	3·01	5·04	71·12	5·73	3·05	0·10	91
Rain beans (*Calliandra Saman*)	14·25	0·50	11·50	60·33	10·87	2·55	0·05	90
Pods from Peru (*Huaranga Prosopis Juliflora*)	11·15	1·47	10·12	59·80	14·01	3·45	0·05	88
Linseed: Turkish	7·55	39·20	20·94	18·63	8·73	4·95	1·35	169
Calcutta	7·60	38·67	18·50	22·36	8·37	4·50	1·05	165
Manchurian	9·35	37·87	21·88	22·58	4·87	3·45	0·20	172
Morocco	—	40·87	—	—	—	—	—	—
Riga	9·45	35·38	22·69	22·20	5·83	4·45	0·70	167
La Plata	7·92	35·12	22·36	24·82	5·82	3·96	0·11	168
Linseed cake:								
English-made, average ...	11·16	9·50	29·50	35·54	9·10	5·20	0·60	133
from Riga seed	16·40	12·73	28·50	31·15	5·77	5·45	0·95	134
American	10·25	9·10	34·63	30·63	9·84	5·55	0·45	140
,, ...	9·95	5·40	31·12	41·22	7·31	5·00	0·05	133
Prime Western Canadian	11·45	7·12	31·81	36·47	8·35	4·80	0·10	134
Bombay	9·60	10·32	30·83	33·86	8·22	7·17	1·92	137

Name.	Water.	Oil.	Albuminoids.	Digestible Carbohydrates.	Woody Fibre.	Mineral Matters.	Sand and Silica.	"Food Units."
Linseed cake: Chinese ...	9·25	10·93	33·12	29·63	10·87	6·20	1·90	140
Calcutta...	9·25	9·83	33·25	34·82	5·70	7·15	2·20	142
Japanese	10·65	8·60	35·88	29·37	8·10	7·40	1·65	140
Galatz	10·75	9·53	37·25	29·51	8·16	4·80	0·15	146
River Plate	10·15	12·60	30·42	26·60	14·43	5·80	0·70	134
Russian	11·70	9·70	35·25	30·35	7·70	5·30	0·60	143
Italian	11·90	11·83	32·62	28·78	9·27	5·60	1·10	140
Chilian	10·90	13·20	30·37	34·38	5·75	5·40	0·25	143
Mexican...	10·85	11·37	28·09	34·63	9·90	5·16	0·62	133
Spanish	11·45	6·77	32·13	32·08	10·27	7·30	1·80	—
Linseed meal: Japanese...	10·95	11·47	33·12	30·91	7·20	6·35	1·10	142
,, ,, ,, ...	10·45	5·77	34·25	34·70	6·93	7·90	2·10	135
,, ,, (extracted)...	13·15	3·03	34·75	34·67	8·75	5·65	0·65	129
Cottonseed: Egyptian ...	8·80	23·93	19·56	21·54	21·16	5·01	0·80	130
Brazilian	9·35	23·20	21·09	24·96	17·05	4·35	0·07	136
Bombay	8·46	19·37	17·95	29·88	19·99	4·35	0·44	123
Burma	8·90	14·63	17·37	31·42	24·13	3·55	0·15	101
South Russian	9·75	24·12	18·66	26·20	17·62	3·65	0·10	133
Chinese	10·85	13·40	18·63	26·64	26·68	3·80	0·05	91
Sudan	10·50	16·50	21·88	26·89	19·88	4·35	0·05	123
Smyrna	9·75	20·87	17·50	25·73	22·25	3·90	0·15	122
Mersyne (Levant) ...	9·90	18·47	15·25	29·41	23·77	3·20	0·05	114
,, (decorticated)	7·55	37·23	30·87	17·15	1·85	5·35	0·10	187
Decorticated cotton cake meal:								
North American Gulf ports	7·85	8·70	46·50	25·45	6·25	5·25	0·05	163
North American Gulf ports (exceptionally rich)	6·65	15·47	51·50	18·43	2·65	5·30	0·05	186
*Atlantic ports ...	7·40	10·16	42·37	25·86	7·06	7·15	0·05	157
Egyptian	11·45	7·46	32·19	30·34	12·06	6·50	0·20	129
Brazilian	9·70	11·06	44·25	22·83	5·76	6·40	0·20	160
,, (another sample)	9·40	12·40	35·62	26·67	10·36	5·55	0·35	147
Peruvian	9·05	13·01	40·96	22·60	5·83	8·55	0·80	158
Shanghai	9·60	13·03	41·12	23·32	6·38	6·55	0·05	158
Decorticated cotton cake:								
Chinese	12·80	9·47	39·88	24·87	6·53	6·45	0·25	152
,, ...	12·60	5·20	37·88	25·94	12·23	6·15	0·20	133
Burma	12·30	10·27	40·84	23·57	6·47	6·55	1·05	151

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* During the past few years an alteration has taken place in the character of decorticated cottonseed cake and meal from the United States. At present, very little cottonseed is being imported into the United Kingdom as cake, the bulk of that arriving here being ground into meal before shipment. More woody fibre is present than formerly. The contracts on which American decorticated cottonseed meals are now usually sold require choice meal to contain at least 40 per cent. albuminoids, prime meal at least 38 per cent. albuminoids, and good meal at least 35·5 per cent. albuminoids, with allowances if the outturn is below the stipulated amount. Texas meal is usually sold on a basis of 52 per cent. oil and albuminoids together.

Name.	Water.	Oil.	Albuminoids.	Digestible Carbohydrates.	Woody Fibre.	Mineral Matters.	Sand and Silica.	"Food Units."
Decorticated cotton cake:								
Russian	7·10	14·50	43·94	22·61	5·20	6·65	0·20	168
Mexican	6·35	10·23	43·12	26·54	7·57	6·19	0·19	160
Bombay	7·50	9·97	35·38	32·00	7·75	7·40	0·65	145
Undecorticated cotton cake:								
English make	13·75	6·56	24·62	29·28	21·19	4·60	0·15	107
„ (another brand)	13·05	6·46	26·31	31·80	17·48	4·90	0·25	114
Egyptian	12·15	5·53	23·01	32·37	21·19	5·75	0·35	104
Chinese	10·15	5·97	29·63	28·88	19·47	5·90	0·40	118
Burma	9·80	5·03	23·38	32·04	23·30	6·45	1·75	103
„	10·50	4·57	18·62	33·33	27·33	5·65	1·30	91
West African	10·30	6·47	23·25	27·45	28·68	3·85	0·30	102
Calcutta	8·95	4·90	21·00	38·45	22·35	4·35	0·25	103
Bombay	9·97	6·05	22·00	34·98	20·94	6·06	1·20	105
Smyrna	10·97	6·78	24·25	33·26	20·11	4·63	0·05	111
Mersyne	11·31	4·39	18·25	35·58	26·56	3·91	0·20	92
West Indian	10·15	6·59	26·25	33·51	18·80	4·70	0·10	116
Brazilian	11·00	5·37	27·12	27·13	24·93	4·45	0·15	108
Undecorticated cottonseed meal (extracted)	9·15	1·26	26·94	28·40	28·45	5·80	0·35	99
Whole seed cotton meal (from cleaned seed)	10·60	23·57	21·62	23·75	16·06	4·40	0·35	137
Cottonseed husks (38·7 per cent. husk in seed)	11·90	0·63	3·50	41·89	39·73	2·35	—	52
Cottonseed pollards ...	10·55	2·07	8·25	40·88	34·50	3·75	0·55	67
Cotton bran	12·25	0·90	2·88	47·74	33·78	2·45	0·10	57
Kapok seed (bastard cotton)	10·85	22·01	22·87	18·65	20·02	5·60	0·10	131
Kapok seed cake	13·80	7·47	26·25	23·19	23·19	6·10	0·38	107
Yellow rape seed	6·50	43·17	24·75	18·30	2·63	4·65	0·75	188
Rape seed, Ferozopore ..	5·70	39·23	22·00	22·45	4·17	6·45	1·65	176
Ravison seed	6·65	23·40	25·50	19·87	10·73	13·85	9·05	142
Rape seed cake:								
Punjaub	7·75	13·27	37·25	28·46	5·90	7·37	2·15	155
Chinese	7·65	10·77	33·13	25·78	9·87	12·80	5·80	135
„	7.45	10·57	27·63	22·75	11·10	20·50	12·95	118
Bessarabian peasant-made	8·10	12·27	33·13	27·32	9·83	9·35	4·15	141
French	8·10	9·27	36·38	29·28	7·97	9·00	2·05	143
Rape seed meal:								
„ „ (extracted)	9·90	3·30	35·42	31·60	11·03	8·75	3·10	128
Jamba (extracted) ...	10·20	2·87	40·25	33·55	8·43	4·70	0·99	141
Jamba cake	7·75	8·40	35·50	30·95	9·00	8·40	2·55	141
Brown East Indian (extracted)	12·01	3·10	35·12	33·00	8·47	8·30	1·80	128
Brown colza cake, Cawnpore	6·55	6·87	35·63	34·15	6·30	10·50	2·75	140
Ravison cake	7·30	7·10	33·12	29·16	12·27	11·05	4·90	130
„ „ Dneiper ...	8·70	12·13	36·75	30·29	6·78	5·35	0·90	152

Name.	Water.	Oil.	Albuminoids.	Digestible Carbohydrates.	Woody Fibre.	Mineral Matters.	Sand and Silica.	"Food Units."
Turnip seed cake, River Plate	12·35	9·60	27·63	33·70	9·97	6·75	0·60	127
Navette cake	11·80	8·47	31·62	28·03	10·53	9·55	2·99	128
Radish cake, Monte Video	7·55	12·90	26·63	32·27	15·45	5·20	0·70	131
Mustard seed meal (extracted)	11·60	2·18	22·50	40·89	16·23	6·60	0·80	—
Mustard seed cake (brown)	12·15	7·73	18·00	39·86	17·61	4·65	0·65	104
"Dodder" cake (Camelina)	8·80	9·43	32·75	27·22	10·20	11·60	4·95	133
"Gold of pleasure" cake (Camelina)	10·25	11·20	35·37	25·64	11·53	6·01	0·80	142
Coco nut	16·13	58·23	5·44	14·26	4·36	1·58	—	159
(Copra) Bombay ...	5·50	67·03	—	—	—	—	—	—
,, South Sea ...	4·15	67·67	8·25	15·70	2·33	1·90	—	206
,, Malabar ...	3·60	70·90	7·88	13·62	2·25	1·75	—	211
Coco-nut cake :								
English	9·01	9·20	21·19	43·04	11·91	5·65	0·30	119
,, (another make)...	8·70	14·96	21·19	40·29	9·51	5·35	0·25	130
Macassar	9·30	16·96	18·75	40·56	8·98	5·45	0·30	130
Bombay	11·90	11·83	23·12	35·70	7·90	9·55	4·10	123
Poonac cake	8·65	15·93	21·12	36·80	10·60	6·90	1·45	129
Coco-nut fibre	11·40	0·90	0·94	54·46	27·90	4·40	0·20	59
Palm kernels	4·90	50·35	9·63	26·58	6·64	1·90	0·05	177
Palm-kernel meal... ...	9·99	12·91	16·18	35·57	21·80	3·55	—	108
(Extracted)	10·69	1·80	19·13	49·06	15·38	3·94	—	101
Buenos Ayres	7·65	25·67	27·25	25·51	8·72	5·20	0·90	158
Palm-kernel cake	9·73	9·14	15·89	41·66	19·74	3·84	—	105
,, shells ...	6·65	7·45	2·25	48·15	32·20	3·30	1·15	56
"Eaba palm" seeds, Demerara	27·50	0·30	4·25	44·48	21·47	2·00	—	58
"Coco meal" (vegetable ivory)	12·25	0·93	4·13	69·92	7·77	5·00	1·05	82
Candle nuts	4·55	63·43	22·13	3·81	2·08	4·00	0·05	218
Candle-nut cake	7·10	16·01	42·87	19·92	5·95	8·15	0·10	167
Euphorbia elastica nuts ...	8·10	39·07	18·25	14·51	16·97	3·10	0·05	158
Calabash seeds, Lagos ...	5·50	52·17	31·37	6·46	0·90	3·60	0·05	215
Acoomoo beans, Lagos ...	4·90	63·03	8·62	12·97	8·53	1·95	—	192
Cuajo nuts (*Myristica Surinamensis*)	3·75	65·23	8·75	10·64	9·68	1·95	0·10	196
Touloucouna nuts, African	9·95	39·40	4·00	38·67	6·48	1·50	0·10	147
"Botter bean" acorn, from Africa (*Quercus Ilex var. Ballota*). Husk 21·9 per cent., kernel 78·1 per cent. Kernels yielded	28·15	5·84	5·12	57·59	1·50	1·80	—	85
"Fulla panza" seed (*Pentaclethra macrophylla*)	8·15	37·01	23·75	25·16	3·48	2·45	0·05	—
Tallow nuts (*Pogo Aleosa*)	4·90	73·40	4·38	11·04	4·53	1·75	—	—
,, ,, ,,	7·90	50·33	7·37	18·80	13·30	2·30	—	—

Name.	Water.	Oil.	Albuminoids.	Digestible Carbohydrates.	Woody Fibre.	Mineral Matters.	Sand and Silica.	"Food Units."
Tallow nuts (*Pogo Aleosa*)	8·45	55·47	7·87	13·24	13·47	1·50	—	—
Para rubber nuts (*Hevea Brasiliensis*)	11·00	24·87	9·63	22·55	30·40	1·55	—	—
Dhomba seed (*Calophyllum inophyllum*)	3·10	74·30	6·88	7·01	6·51	2·20	—	210
Dhomba meal (extracted)	10·00	20·23	20·75	29·74	11·53	7·75	2·10	132
Coulby nuts	4·82	61·70	7·82	17·30	5·09	3·27	0·05	191
Lamy nuts...	7·87	46·80	4·51	32·85	6·00	1·97	0·10	161
Cohune nuts (kernels) ...	6·25	69·71	7·50	12·62	2·07	1·85	—	206
Pignolia kernels	7·25	45·50	37·25	4·98	0·47	4·55	0·05	212
Shea nuts, West African ...	5·65	48·80	7·38	32·74	2·68	2·75	0·05	173
„ „ „	5·80	52·67	9·38	26·15	3·25	2·75	—	181
Shea-nut meal	12·70	4·73	12·37	57·67	6·48	6·05	0·65	100
Sesbania macrocarpus, from Colorado River	10·80	6·03	37·25	31·29	11·08	3·55	0·30	139
"Chakore" seed (*Cassia Tora*)	12·10	6·60	17·25	49·77	9·23	5·05	0·75	102
Niger seed cake	8·90	14·03	34·06	21·80	9·26	11·95	3·15	142
„ and Kurdee (safflower) cake	8·15	8·73	29·01	24·96	19·40	9·75	3·45	119
Sesame seed, Cyprus ...	—	53·80	—	—	—	—	—	—
„ cake, Burma ...	7·20	17·80	35·88	22·44	4·43	12·25	1·95	156
„ „ Brazilian ...	6·50	30·72	34·13	16·77	3·68	8·20	0·64	179
„ „ Egyptian ...	8·35	8·20	44·75	18·82	5·53	14·35	4·95	151
"Sim-Sim" cake, East Africa (Sesame)	8·55	17·13	38·88	21·22	3·97	10·25	1·25	161
Till cake, Akola (India Central Provinces)	8·40	8·93	38·87	25·50	3·50	14·80	2·75	145
Gingelly and Niger seed cake	7·25	12·53	34·63	19·42	12·97	13·20	5·25	137
Gingelly and Kurdee cake	8·35	10·60	26·12	23·76	21·27	9·90	3·10	115
Earth nuts, Rangoon ...	7·90	39·03	20·50	12·84	17·58	2·15	0·10	161
Ground nuts:								
Decorticated Chinese ...	5·35	45·73	25·88	19·64	1·25	2·15	0·05	199
„ W. African	7·00	44·03	27·63	17·64	1·00	2·70	—	197
Ground-nut cake, decorticated, Rangoon	8·25	5·53	48·00	28·35	1·67	8·20	4·05	162
Ground-nut cake, decorticated, River Plate	9·15	16·60	44·25	21·09	4·46	4·45	0·55	173
Pea-nut cake, American ...	6·10	16·90	40·00	27·90	4·70	4·40	0·80	170
Ground-nut cake, undecorticated, Burma	10·10	9·33	28·50	25·20	22·47	4·40	1·00	120
Ground-nut cake, undecorticated, Macassar	8·85	9·43	31·63	22·94	17·73	9·42	4·92	126
Arichide shells	7·85	5·03	7·38	20·94	54·90	3·90	2·20	52
Earth nut husks (hand-picked)	15·00	1·30	6·01	20·04	54·55	3·10	0·45	38
Earth nut and Kurdee cake	11·80	8·46	38·62	23·14	11·63	6·35	2·20	140

Name.	Water.	Oil.	Albuminoids.	Digestible Carbohydrates.	Woody Fibre.	Mineral Matters.	Sand and Silica.	"Food Units."
Earth nut and Kurdee cake (another sample)	8·15	8·63	30·87	23·21	21·13	8·01	2·10	122
Ground nut and Niger cake	7·70	9·03	37·37	24·83	12·27	8·80	2·30	141
Kurdee cake (Safflower) ...	8·55	9·73	20·25	25·12	32·95	3·40	0·70	100
,, ,, decorticated	11·60	7·70	47·88	19·72	6·20	6·90	1·65	158
Sunflower seed cake ...	7·10	7·43	19·01	28·93	30·03	7·50	2·65	95
Husked	7·75	10·03	37·00	21·14	16·53	7·55	2·20	139
Egyptian	9·95	7·23	16·06	30·71	24·70	11·35	5·75	89
South Russian, peasant made	7·27	18·47	33·19	19·34	14·83	6·90	1·60	148
Odessa, hydraulic ...	7·75	8·68	38·38	22·46	16·03	6·70	0·95	140
Marrotty seed meal, extracted (*Hydnocarpus venenatus*), poisonous	7·05	4·30	23·00	27·55	30·70	7·40	1·10	—
Castor-oil seeds, poisonous	7·31	43·96	17·75	7·76	20·20	3·02	—	—
Cottonseed meal (extracted)	11·75	1·10	30·62	13·96	36·52	6·05	—	—
" Nut residue " from mixed Sapotaceæ, South Sea Islands	9·90	7·96	14·57	49·54	14·43	3·60	1·50	106
Canary-seed cake	9·54	19·80	17·35	38·50	8·67	6·14	2·19	131
Mahwa (Bassia) cake (unfit for feeding)	8·45	16·53	15·25	48·21	5·16	6·40	1·45	—
Mahwa flowers	12·75	1·57	6·00	65·85	3·13	10·70	7·75	—
Illipé meal...	7·60	30·16	8·63	42·94	5·77	4·90	1·10	—
,, ,, (extracted) ...	17·90	10·27	10·25	45·76	9·97	5·85	1·10	—
Shea-nut meal	7·85	20·30	11·13	48·54	6·68	5·50	0·90	—
" Olive-kernel meal " ...	8·40	2·23	1·12	43·08	43·41	1·76	0·65	—
Olive-nut refuse	10·65	17·33	7·75	26·91	34·51	2·85	—	—
,, ,, ...	12·80	4·33	6·94	38·57	30·46	6·90	4·50	—
Coffee bran	9·45	0·77	2·37	24·76	61·75	0·90	0·10	32
Cacao, Guayaquil... ...	3·55	47·20	15·38	27·39	2·53	3·95	0·15	183
,, shells	9·30	3·83	18·81	46·01	13·85	8·20	0·85	102
Hop seeds	11·35	18·63	30·37	15·90	16·60	7·15	2·30	138
,, meal	7·15	9·93	27·50	31·90	16·97	6·55	1·75	125
Arrowroot from Formosa (natural condition)	63·00	0·23	2·12	32·33	0·87	1·45	—	38
Sago flour, Borneo ...	15·70	0·20	1·37	75·61	2·17	4·95	4·25	79
,, ,, 1st quality ...	11·70	0·13	0·13	87·56	0·13	0·35	0·10	88
,, ,, 2nd quality ...	16·30	0·17	1·12	76·91	3·30	2·20	1·60	80
Farina middlings	13·55	0·63	1·88	80·21	1·58	2·15	0·10	86
Tapioca flour, Ampas ...	12·70	0·23	0·88	80·47	4·87	0·85	—	83
,, ,, Penang ...	11·80	1·27	3·00	78·73	0·60	4·60	3·65	89
,, ,, Low Java...	13·80	0·43	1·37	74·08	5·47	4·85	2·10	79
,, -seed pearl ...	13·50	0·20	0·12	85·96	0·07	0·15	—	87
Manioc-roots meal, Java	11·70	0·70	2·63	81·62	1·65	1·70	0·10	90
Buckwheat	10·80	2·45	10·13	67·29	7·73	1·60	0·05	99
Poppy-seed cake	10·15	11·43	35·38	20·04	7·90	15·10	3·15	—
,, meal	9·95	12·13	39·50	16·24	9·43	12·75	1·40	—

Name.	Water.	Oil.	Albuminoids.	Digestible Carbohydrates.	Woody Fibre.	Mineral Matters.	Sand and Silica.	' Food Units."
Hemp-seed cake	12·55	8·30	32·38	16·02	22·90	7·85	2·10	118
" meal, Russian	11·75	1·73	34·75	16·82	25·70	9·25	3·15	108
" cake, Manchurian	7·40	9·83	30·88	20·32	25·07	6·50	2·20	122
" Kandy cake " (*Pongamia Glabra*)	10·45	13·67	27·62	42·18	1·98	3·10	0·10	
Treacle	22·22			71·28		5·80		71
Dried brewer's grains ...	10·01	6·93	19·50	42·31	17·60	3·65	1·75	108
distillery grains	6·05	10·73	32·75	37·15	10·67	2·65	0·60	146
Distillery grains sediment	9·40	13·37	34·12	30·19	10·67	2·25	1·10	149
Malt refuse	51·42	1·50	7·37	37·80	1·26	0·65	0·15	60
" " cake	42·55	3·80	11·50	34·92	5·03	2·20	1·01	73
Distillery meal	5·10	23·50	25·19	33·87	11·33	1·01	0·35	156
" grains	73·98	2·15	5·88	12·35	4·86	0·78	0·26	32
Pressed yeast	71·70	0·85	16·44	8·10	—	2·90	—	51
Malt, culms	9·80	1·03	26·28	45·96	11·08	5·85	1·55	114
Combings, No. 2	10·10	1·55	21·25	45·28	15·17	6·65	2·90	112
" No. 3	10·75	1·72	22·12	45·56	13·90	5·95	2·01	115
" Crawtaes " (Achenes of *Ranunculus repens*) poisonous	10·80	13·70	11·30	18·25	36·05	9·70	3·60	
" Distilled grape seeds " ...	8·60	10·72	10·06	33·76	31·54	5·32		86
" Apple offal "	9·50	5·93	9·50	48·15	19·27	7·65	4·15	87
" Tomato meal "	7·25	15·62	27·00	14·93	30·05	5·15	0·70	121
Beet pulp cake	12·25	2·77	6·88	55·81	18·59	3·70	0·35	80
Dried beet pulp	11·80	0·83	8·75	58·92	16·45	3·25	0·05	83
Beet fodder, Spanish ...	12·00	0·60	8·87	61·52	10·21	6·80	0·05	85
Megass meal	11·10	1·47	2·00	51·88	30·10	3·45	2·25	60
Peat dust	21·15	2·90	6·50	43·65	22·80	3·00	1·99	
Carui chaff	13·50	4·03	8·88	37·60	24·59	11·40	0·55	
Canadian stock feed ...	12·50	11·40	12·25	34·02	16·63	13·20	8·30	
Meadow grass	70·48	0·83	2·95	14·14	9·54	2·06		23
Ensilage from same, salted	68·74	2·35	3·04	11·10	9·90	4·87		24
Meadow hay (Plot 7, Saltney)	14·30	1·48	9·32	41·28	26·83	6·79	1·42	68
Clover hay (Plot 15 Saltney)	16·68	0·86	11·75	37·39	25·33	7·99	1·99	68
Swedes (average of fourteen analyses)	90·42	0·20	0·91	6·61	1·29	0·57		9
Straw, wheat	13·81	1·47	2·96	30·29	47·07	4·40		41
barley	14·65	1·45	3·25	35·35	41·00	4·30		47
" oat ...	15·15	1·52	4·04	38·47	35·64	5·18		52
Turnips	91·85	0·20	1·05	5·25	0·95	0·70		8
Mangels	87·89	0·17	1·32	8·63	1·06	0·93		12
Potatoes	75·80	0·20	2·08	19·90	1·10	0·92		26
Carrots	87·25	0·20	1·25	8·80	1·60	0·90		12

Nitrogenous Constituents of Hops. A. C. Chapman. (*J. Chem. Soc.*, 1914, **105**, 1895-1907.)—In the samples analysed the total nitrogen ranged from 1·72 to 4 per cent., the majority of the numbers falling between 2 and 3·5 per cent. The nitrogen soluble after boiling the hops for about an hour varied from 0·44 to 0·9 per cent. The following are some typical values:

	Kent.	Bohemian.	Oregon.
	Per cent.	Per cent.	Per cent.
Soluble nitrogen	0·74	0·54	0·44
Insoluble „	2·07	1·36	1·56
Total nitrogen	2·81	1·90	2·00

The soluble nitrogen was found to be apportioned as follows in average extracts from English, American and German hops:

	Per cent. in the Dry Hops.
Soluble proteins and albumoses	0·046
Ammonium salts and amides	0·100
Amino substances	0·095
Bases and unclassified nitrogen precipitated by phosphotungstic acid	0·218
Unclassified nitrogen not precipitated by phosphotungstic acid (by difference)	0·496

Four methods were adopted for the extraction of the hops (ANALYST, 1913, **38**, 459).

First Method.—The concentrated aqueous extract prepared by extracting fresh hops first with alcohol and then with water was treated with a solution of basic lead acetate, filtered, and the lead removed from the filtrate with hydrogen sulphide, after which a slight excess of phosphotungstic acid was added. The precipitate, which consisted chiefly of potassium phosphotungstate, was cautiously ground in small quantities with barium hydroxide and a little water; the barium being afterwards separated from the filtrate by carbon dioxide. After further treatment histidine was identified and probably arginine in this fraction, and in the filtrate from the two purine precipitates were found choline and betaine. The phosphotungstic acid filtrate was found to contain *l*-asparagine having a specific rotatory power $[\alpha]_D - 5{\cdot}08$, and in the basic lead acetate precipitate first obtained aspartic acid was identified, this substance being separated in the form of the blue needle-shaped crystals of its copper salt. Neither glutamine nor glutamic acid was found in any of the fractions in which they would have been likely to appear.

Second Method.—The hops were finely minced in a sausage machine, and were then mixed with twice their weight of slaked lime. Water was added, and the mixture heated nearly to boiling, after which it was evaporated to dryness at a low temperature on the water-bath. The dry mass was then powdered and extracted with much alcohol, which was then made slightly acid and the alcohol distilled off, the residue being dissolved in water. This solution gave precipitates with Mayer's reagent, picric acid, bismuth potassium iodide, and other base reagents. Owing to the presence of much non-nitrogenous organic matter and inorganic salts, the phosphotungstic acid separation had to be resorted to. In this fraction was identified adenine, which was separated as picrate, and in the filtrate from this substance hypoxanthine was found, but not xanthine. Choline and betaine were again obtained and identified.

Third Method.—Ammoniacal amyl alcohol was the next solvent employed, but after the separation of the purines, little else but adenine was obtained. Choline and betaine were again found.

Fourth Method.—A quantity of aqueous hop extract was warmed on the bath with alcohol mixed with half its volume of glacial acetic acid. The extract was finally distilled with steam, when a quantity of resin separated. The liquid remaining in the flask after the removal of the alcohol was evaporated repeatedly with water until the greater part of the acetic acid had gone off. The aqueous liquid was then made slightly acid, and thoroughly extracted with the following immiscible solvents in the order named: (1) benzene, (2) chloroform, (3) ether. The liquid was then made slightly alkaline, and the extractions continued with (4) benzene, (5) chloroform, (6) amyl alcohol. No indication of morphine or any similar alkaloid could be obtained in any of the first three cases. Alkaline benzene yielded a trace of a dark-coloured oil with an odour strongly resembling coniine. It gave a slight curdy precipitate with Mayer's reagent. Alkaline chloroform yielded no evidence of morphine, and alkaline amyl alcohol gave a fair amount of residue, consisting of impure hypoxanthine.

It was found that hops always contained very appreciable amounts of potassium nitrate. Fractional dialysis of aqueous hop extracts did not give encouraging results. The author concludes that it may safely be assumed that the cultivated hops of commerce are free from morphine, or contain at most traces without physiological or other significance (ANALYST, *loc. cit.*). H. F. E. H.

Estimation of Mercuric Iodide in Tablets. A. W. Bender. (*J. Ind. and Eng. Chem.*, 1914, **6**, 753-754.)—The powdered tablets (containing 1 to 2 grains of mercuric iodide) are heated on a sand-bath beneath a reflux condenser with 20 c.c. of hydrochloric acid (1 : 1) and about 0·5 grm. of potassium chlorate until all mercuric iodide is dissolved. The solution is cooled and diluted to about 100 c.c., and a current of air passed through it to remove chlorine compounds. It is then filtered, the residue washed by decantation, the filtrate and washings made alkaline with ammonia, and the mercury precipitated with hydrogen sulphide. After standing for a few hours, the mercuric sulphide is collected in a tared Gooch crucible, washed with water and alcohol, dried at 100° C., and weighed. C. A. M.

Composition and Adulteration of Rose Oil. N. Petkow. (*Zeitsch. f. offentl. Chem.*, 1914, 149; through *Pharm. J.*, 1914, **93**, 349.)—Adulteration of rose oil with geranium oil may be recognised by the higher saponification number and refractive index, and some adulterators now add artificial stearoptenes, ceril, etc., to conceal the sophistication. The author has investigated these compounds and records their constants. In addition to the usual constants used in examining rose oil—specific gravity, optical rotation, acid number, ester number, etc.—the author has determined the iodine number and refractive index, and lays special stress on the value of the latter figure. The iodine number of several Indian (or Turkish) geranium oils varied between 230 and 250, of the French oils between 170 and 176·3, and of true rose oils between 170·8 and 189·1. The refractive index of Palmarosa oil and all ethereal oils used for adulteration is over 70, and of French geranium oil higher than 60, while the figure for genuine rose oil is considerably lower, between 44 and 48. Comparison of the results with fifteen samples of rose oil shows that the detection of adulteration, if skilfully carried out, is often extremely difficult. A complete determination of all the constants is necessary in order to say with certainty whether a given oil is genuine or not. In Bulgaria a "rose-concrete" is prepared by extraction with benzine. This contains, besides oil of rose, the colouring matter, wax, resin, etc., of the flowers. About 300,000 kilos of flowers are worked up in this way in Bulgaria every year. The "rose-concrete" forms a yellowish soft mass with a very agreeable rose-like odour. The constants of this product have also been determined and are recorded in the original paper.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Studies in Bacterial Metabolism in Milk. A. I. Kendall, A. A. Day, and A. W. Walker. (*J. Amer. Chem. Soc.*, 1914, **36**, 1937-1962.)—The observations recorded below were made for the purpose of determining the nature and extent of the changes brought about by the growth of various important types of bacteria in sterile milk.

Typhoid-dysentery-alcaligenes Group.—*B. alcaligenes*, the Flexner and Shiga types of the dysentery bacillus, and *B. typhosus* produce no marked alterations in the appearance of milk, or any noteworthy changes in the composition as indicated by changes in reaction and ammonia production. The presence of even large numbers of these organisms could not be detected by chemical methods during the time when this milk would be saleable. The production of an initial acidity followed by an alkaline reaction, which is a well-known characteristic of the dysentery bacillus, may be explained by the chemical changes brought about by the organisms. The explanation of the permanent acidity exhibited by cultures of typhoid bacilli in milk has not been definitely determined.

The Intermediate or Paratyphoid Group.—During the early days of incubation the composition of milk is not materially changed by the growth of *B. paratyphosus*, *B. paratyphosus β*, *B. icteroides*, or the Morgan bacillus. The initial acidity exhibited by all the members of this group is due to the fermentation of the dextrose-like substance which appears to be a normal constituent of fresh milk. *B. paratyphosus α* reacts like *B. typhosus*—that is, it produces a permanent acidity. The Morgan bacillus is more proteolytic than are the other members of the group.

Coli-proteus-cloacæ Group.—*B. coli* and *B. cloacæ* ferment lactose vigorously, without producing noticeable quantities of gas, but the lactose appears to protect the milk proteins from decomposition. *B. proteus* does not attack lactose, but decomposes the milk proteins with the formation of ammonia.

Subtilis-mesentericus Group and B. pyocyaneus.—The milk proteins are readily decomposed by these three organisms, the quantity of ammonia produced being much greater than that formed by the ordinary pathogenic bacteria. In the case of *B. subtilis*, the milk has a progressively alkaline reaction, with *B. pyocyaneus* there is a transient initial acidity, whilst *B. mesentericus* produces a progressive acidity. *B. pyocyaneus* gives a green coloration in milk even after only twenty-four hours' growth.

B. diphtheriæ, B. suipestifer, Vibrio choleræ, and B. tuberculosis.—The diphtheria bacillus produces no visible change in milk even after three weeks' growth at blood-heat, and the changes in composition are so slight as to escape detection by chemical methods. The cholera vibrio produces an acid coagulation of the milk after three days' growth, due to the fermentation of the lactose; there is but little coincident decomposition of the proteins. Human tubercle bacillus grows rapidly in milk, producing a moderate breakdown of the proteins and a progressive alkaline reaction; at the end of three weeks' incubation the milk has a decided brown colour, and is almost opalescent.

The Coccal Group.—Streptococcus pyogenes, Staphylococcus aureus, and *Micrococcus zymogenes* produce an acid coagulation of the milk after about three days. The amount of acid formed varies with the organism, *Streptococcus pyogenes* yielding relatively little acid, *Staphylococcus pyogenes* and *Micrococcus zymogenes* producing considerable amounts. *Staphylococcus pyogenes* also produces less ammonia than does *Staphylococcus aureus* and *Micrococcus zymogenes*, and milk cultures of the two latter organisms exhibit a considerable accumulation of clear serum after coagulation is complete; it is not known whether this is due to peptonisation of the casein or to mechanical contraction of the protein. *Micrococcus melitensis* grows slowly in milk and produces no visible change in the medium.

B. typhosus, B. paratyphosus α and β, *B. coli*, and the tubercle bacillus "W" do not exhibit any marked differences in their nitrogen metabolism as measured by ammonia formation, or in their reactions towards various indicators when they are grown under similar conditions in whole milk, skimmed milk, or cream. The permanent acid reaction, which is a feature of the growth of *B. typhosus*, and *B. paratyphosus* α, in whole milk, is also produced in skimmed milk and cream. The initial acidity followed by alkalinity, which is characteristic of *B. typhosus* and *B. paratyphosus* β in milk, is also observed in cream. *B. proteus* is more proteolytic in its action in cream than it is in whole milk or skimmed milk, whilst *B. pyocyaneus* is less proteolytic in cream. W. P. S.

Estimation of Small Quantities of Lead, Particularly in Urine in Cases of Lead Poisoning. G. Meillere. (*J. Pharm. Chim.*, 1914, **10**, 225-231.)—Small amounts of lead may be separated from a large volume of liquid by adding a quantity of copper sulphate and precipitating the two metals as sulphides; the mixed sulphides are then dissolved, and the lead is deposited electrolytically on a small electrode. For the estimation of lead in urine, the quantity voided during twenty-four hours is acidified with 1 per cent. of its volume of hydrochloric acid, 1 grm. of copper sulphate is added, and a slow current of hydrogen sulphide is passed through the solution. When precipitation is complete, the solution is heated for a short time on a water-bath, the precipitate is then collected on a filter, washed, and dissolved in 10 c.c. of nitric acid. The filter is washed with hot water, the acid solution and washings are evaporated to dryness, and the residue is ignited until the cupric nitrate has been converted into cupric oxide; any traces of organic matter and sulphur are thus destroyed. This residue is now dissolved in 6 c.c. of concentrated nitric acid, the solution is diluted to 100 c.c., and submitted to electrolysis. Small platinum electrodes are employed, and with a current of 2 volts and of such ampèrage that there is a just perceptible evolution of gas from the anode, the whole of the lead is deposited as peroxide within twenty-four hours. The deposited lead peroxide may be weighed when an appreciable quantity is present, and in any case, the deposit should be dissolved in nitric acid containing oxalic acid, and the lead identified by the application of suitable tests to the solution.

W. P. S.

ORGANIC ANALYSIS.

Estimation of Casein by Means of Ferric Ammonium Sulphate. H. V. Arny and H. H. Schaefer. (*J. Ind. and Eng. Chem.*, 1914, **6**, 748-751.)—About 5 grms. of the milk are mixed with 5 c.c. of ferric ammonium sulphate solution (48·2224 grms. per litre), and the mixture filtered after standing for an hour. The precipitated casein is washed until free from iron, the filtrate and washings (about 200 c.c.) digested for thirty minutes at 40° C. in a stoppered flask, with 3 c.c. of 31 per cent. hydrochloric acid and 2 grms. of potassium iodide, and the free iodine titrated with $\frac{N}{50}$ thiosulphate solution (1 c.c. = 0·00964 grm. of iron). Each c.c. of the standard iron ammonium sulphate solution precipitates 0·05934 grm. of casein from milk, and the results thus obtained within thirty-five minutes agree closely with the amounts calculated from the nitrogen estimated by the Kjeldahl-Gunning method. In test experiments with dry casein the figures did not agree so closely as in the case of milk, and the average equivalent of 1 c.c. of the iron solution in grms. of casein was lower (0·0479 grm.).

C. A. M.

Quantity and Character of Creosote in Well-Preserved Timbers. G. Alleman. (*Proc. Amer. Wood Preservers' Assoc.*, 1914, 88-96; through *J. Soc. Chem. Ind.*, 1914, **33**, 832.)—The creosote oils extracted from timber which had been in use many years showed an average of 32·9 per cent. of distillate below 270° C. and 66·8 per cent. of oils of high boiling-point. The defects of most modern creosote preservatives are deficiency in basic oils of high boiling-point and the substitution for these of tar or other viscous substances. Under proper conditions of distillation a stable, heavy creosote oil (sp. gr. 1·10) could be manufactured from coal tar, containing nothing which boils below 210° C.; for general purposes not more than 50 per cent. of the total should distil below 315° C., and for wood-paving blocks not more than 35 per cent. The qualities demanded are penetrating power, stability, and preservative and water-proofing value. All these are possessed in a high degree by pure heavy creosote oil. Tar has a very low penetrating power, even when injected at a high temperature, and wood treated with a mixture shows a separation of the components, oil, tar, and carbon, at the margins of the close-grain rings. As regards stability, losses occur owing to volatilisation, extraction by water, and crystallisation in wood impregnated with light creosote oils. Preservative value may depend on direct antiseptic action or permanent adhesion of a stable oil-coating to the walls of the minutest cells; external filming without penetration is of little account. In the light oils the antiseptic constituents predominate, but are volatile and largely soluble in water. The heavy oils are sufficiently rich in the higher homologues of cresols, both these and the protective oils being far more stable. Waterproofing also depends on the complete coating of the cell walls with an oily deposit which excludes moisture.

Report of the Committee on Wood Preservatives (Creosote Oil). (*Proc. Amer. Wood Preservers' Assoc.*, 1914, 58-76; through *J. Soc. Chem. Ind.*, 1914, **33**, 832.)—The definition of creosote oil is revised, and comprises all distillate oils boiling between 200° and 400° C., obtained by direct distillation from tars consisting principally of compounds of the aromatic series and containing well-defined amounts of phenoloids. The most important tests are—For moisture, specific gravity, and distillation. Additional tests are—For free carbon, tar acids, sulphonation; the Hempel distillation test, refractive indices, and specific gravities of the fractions. These tests are described in detail together with the apparatus employed. All temperature observations must be made with a standard thermometer of specified dimensions, length of stem from 0° to 400° C., 295 mm., bulb 14 mm. long and 6 mm. in diameter. Readings to be uncorrected. No recommendations have yet been formulated as to standard specifications for creosote oils, but the specifications enforced by several consumers' associations are reprinted collectively for comparison.

Absorption of Dextrose by Bone-Black. H. A. Morton. (*J. Amer. Chem. Soc.*, 1914, **36**, 1832-1838.)—Purified bone-black absorbs dextrose very slowly at ordinary temperatures, but more rapidly at 80° C. In experiments with solutions of dextrose ranging from 4·63 to 50·58 per cent., equilibrium was reached at 80° C. after six to ten days. The ratios of the weight of sugar per grm. of solution to that per grm. of bone-black were found to be practically the same (mean = 0·46) whatever the concentration. Hence, when equilibrium had been reached, the amount of sugar in 1 grm. of bone-black was more than twice that contained in 1 grm. of solution. It was found that the bone-black also absorbed part of the water from the dextrose solutions.

C. A. M.

Leaf and Twig Oils of Cuban and Long-leaf Pines and Cone Oil of Long-leaf Pine. A. W. Schorger. (*J. Ind. and Eng. Chem.*, 1914, **6**, 723-727.)—These oils had the following percentage composition :

	Cuban Pine (*Pinus hetero-phylla*).	Long-leaf Pine (*Pinus palustris*).		
	Leaf and Twig.	Leaf and Twig.	Leaf.	Cone.
Furfural	trace	trace	trace	trace
l-*a*-pinene	4	8 to 9	2	39 to 40
l-camphene	10	13 to 14	12 to 13	12
l-*β*-pinene	35 to 36	44	50	25
Dipentene	8	5·0	5	6 to 7
Bornyl esters (as acetate)	3·5	2·4	2	1·4
Free alcohol (*a*-*l*-borneol)	11·4	10	9·8	7·6
d-cadinene	18 to 19	10 to 11	11	1 to 2
Loss by polymerisation, etc.	9	6	7·5	6·5

Their physical and chemical constants were as follows :

	Cuban Pine.	Long-leaf Pine.	
	Leaf and Twig Oils.	Leaf and Twig Oils.	Cone Oil.
Sp. gr. at 15° C.	0·8877 to 0·8894	0·8829 to 0·8849	0·8756
$[N]_{D\ 15^\circ C.}$	1·4845 to 1·4869	1·4818 to 1·4825	1·4760
Rotation $[a]_{D\ 28^\circ C.}$	$-32·09°$ to $-35·67°$	$-26·38°$ to $-30·49°$	$-9·22°$
Acid value	0·65 to 0·75	0·55 to 0·73	0·42
Ester value...	9·73 to 10·54	6·05 to 7·22	3·95
Ester value after acetylation ...	46·26 to 53·81	36·53 to 46·37	31·07

Fractional Distillation.	Per Cent.	Per Cent.	Per Cent.
155 to 160° C.	—	—	11·0
$\left.\begin{matrix}158\\159\end{matrix}\right\}$ to 165	31·0	38·0	61·0
165 to 170	18·0	25·0	11·0
170 to 180	7·0	5·0	3·5
180 to $\left\{\begin{matrix}195·5\\198·5\end{matrix}\right\}$	3·0	2·0	1·5
$\left.\begin{matrix}195·5\\198·5\end{matrix}\right\}$ to 250	16·0	16·0	8·3
250 to $\left\{\begin{matrix}275\\280\end{matrix}\right\}$	18·5	10·7	1·4
Residue	6·0	3·0	2·3

C. A. M.

Some Natural Indicators. H. W. Brubaker. (*J. Amer. Chem. Soc.*, 1914, **36**, 1925-1928.)—The colouring matter of the rugosa rose (*Rosa rugosa*) gives a green coloration with alkalis, a red coloration with acids, and is colourless at the intermediate point. It may be separated by triturating the petals with sand and 95 per cent. alcohol, filtering the extract, allowing the filtrate to evaporate spontaneously, and taking up the residue with water. Addition of alcohol to bring the alcoholic strength to 50 per cent. will preserve the solution. The indicator behaves like phenolphthaleïn towards carbon dioxide. In the form of test-paper it is sensitive to 1 part of sodium hydroxide in 25,000 parts of water. The colouring matters of the perennial pea (*Lathyrus latifolius*), purple vetch and iris, behave in an analogous manner, and appear to be closely related to the colouring matters of hollyhocks and dahlias. The petals of most flowers are changed in colour by alkalis, being reconverted to the original shade or changed to red by acids. The yellows, however, are unaffected by either acid or alkali, while the whites (*e.g.*, clover, white rose, pansy, geranium) are changed to yellow by alkalis, and made white again by acids. The reds and purples are changed to green or greenish-blue by alkali, while they are restored to their original colour or are made a brighter red by acids. As a rule these natural colouring matters are acid, or in some cases neutral. C. A. M.

Specific Heat of California Petroleums. H. E. Wales. (*J. Ind. and Eng. Chem.*, 1914, **6**, 727-728.)—The average specific heat of California petroleums is about 0·45, the extreme values recorded for water-free samples being 0·400 and 0·483, the lower values all corresponding to petroleum of high asphalt content, the higher ones to petroleum of low asphalt content. Wet samples naturally have a higher specific heat than others similar in all respects save water content, but since it is the heavier, more asphaltic oils that tend to hold most water, no specific heat higher than 0·502 has been found. G. C. J.

INORGANIC ANALYSIS.

Detection of Antimony. J. Petersen. (*Zeitsch. anorg. Chem.*, 1914, **88**, 108.)—The method depends on the formation of the sparingly soluble antimoniate of sodium, and can be applied direct to the mixed sulphides of antimony, arsenic and tin, as separated in the ordinary course of qualitative analysis. The sulphides, mixed with 10 to 15 c.c. of water, are treated with excess of sodium peroxide, the mixture being finally heated to boiling and then filtered hot if stannic acid separates, as may be the case in presence of large quantities of tin. On cooling, the filtered or unfiltered liquid, as the case may be, will deposit crystals of sodium antimoniate if antimony is present in sufficient amount. G. C. J.

Rational Analysis of Clays. J. Burian and J. Juránek. (*Chemické listy*, 1912 [1-5]. *Silikat-Zeitsch.*, 1914, **2**, 12-13; through *J. Soc. Chem. Ind.*, 1914, **33**, 963.)—Experiments were made on the effect of the concentration of, and the time of digestion at the boiling-point with sulphuric acid on the decomposition of the clay substance; the effect of sulphuric acid at different concentrations on felspars and mica; the effect of lyes on felspars; the effect of hydrochloric acid on the clay substance and felspars; and the oxidation of organic substances in clays, by chromic acid. The following improved process was devised: 3 grms. of clay, dried at 120° C., are heated in a covered porcelain dish for 6 to 8 hours with 25 c.c. of concentrated sulphuric acid, till white fumes appear. When cool, the liquid is diluted with hot water, and decanted three times, the decanted liquid being collected in a 2-litre cylinder. The residue is boiled for 5 minutes with 25 c.c. of a mixture of equal parts of 5 per cent. NaOH, and 5 per cent Na_2CO_3 solution, then diluted with water, the residue washed twice by decantation, and boiled for 5 minutes with 10 c.c. of concentrated hydrochloric acid. The treatments with alkali and acid are repeated, the decanted liquid being united with that collected previously, and the residue then washed on a filter with dilute hydrochloric acid. The combined decanted liquids are allowed to stand twelve hours, the clear portion siphoned off, and the residue twice boiled for five minutes with alkali and acid, and brought on to the same filter. After igniting and weighing, the residue is decomposed with hydrofluoric and sulphuric acids, and the quantity of felspar in it calculated from the alumina. Any considerable quantity of organic matter can be removed by gradually heating the clay in the presence of dilute sulphuric acid (1 : 2) and adding chromium trioxide. The oxidation is completed in five minutes, and after the clay has settled the green liquid is decanted and the residue washed with hot water, and the rational analysis carried out as above.

Analysis of Copper, Tin, and Silicon Alloys. E. D. Koepping. (*J. Ind. and Eng. Chem.*, 1914, **6**, 695-696.)—On account of their insolubility in nitric acid and for other reasons, the choice of methods for the analysis of these alloys is limited. The following method has been found satisfactory: The alloy (0·5 grm.) is decomposed by treatment with 20 c.c. water, 15 c.c. hydrochloric acid, 5 c.c. nitric acid, and boiling. After addition of 20 c.c. sulphuric acid, the mixture is evaporated until fumes arise, and is then cooled and diluted with 75 c.c. water. When all sulphates have been brought into solution by boiling, the silica is filtered off and washed with hot water until the filtrate and washings nearly fill a 200 c.c. flask. The silica is ignited and weighed, and the filtrate and washings are divided into two equal portions. One of these is rendered ammoniacal, then acidified with nitric acid, and digested on the hot plate for an hour. The stannic acid, which should be white, is filtered off, washed, and rejected. The filtrate and washings are evaporated with 5 c.c. sulphuric acid until fumes arise, cooled, diluted, neutralised with ammonia, made acid with 7 c.c. of a mixture of 2 parts 50 per cent. nitric acid and 1 part 50 per cent. sulphuric acid, washed into a 150 c.c. beaker, and the copper deposited electrolytically.

In the other half of the filtrate from the silica tin is estimated by the following modification of Low's method: To the solution contained in a 400 c.c. flask 0·15 grm. powdered antimony dissolved in 5 c.c. sulphuric acid, 35 c.c. of hydrochloric acid and 1 grm. of iron filings are added. The mixture is boiled until the iron is nearly all dissolved, and the flask is then closed with a three-hole stopper, carrying a solid plug in one hole, a right-angled bend dipping into the liquid, and connected to a source of carbon dioxide in another, whilst the third hole accommodates an exit tube which connects to a small flask holding a saturated solution of sodium bicarbonate which acts as a seal. Boiling is continued, whilst the current of carbon dioxide is passed, until all iron is in solution and reduction complete. This may require twenty to thirty minutes. Without stopping the current of gas, the contents of the flask are cooled, 5 c.c. starch solution introduced through a funnel substituted momentarily for the solid plug, and the tip of a burette containing $\frac{N}{10}$ iodine solution is finally substituted for the funnel. The current of gas is now stopped, and the tin titrated in the usual manner with $\frac{N}{10}$ iodine, which should be standardised against pure tin. G. C. J.

Influence of Weather Conditions upon the Amounts of Nitric Acid and of Nitrous Acid in the Rainfall. V. G. Anderson. (*British Association Report*, 1914, No. 143.)—Daily determinations of the amounts of nitric acid and of nitrous acid in the rainfall near Melbourne have been made since November 1, 1912. The results to February 28, 1914, when correlated with meteorological data for Melbourne and daily isobaric charts of Australia, reveal the existence of a relation between weather conditions and the amounts of nitrogen acids in rain-water.

The concentration of nitric acid reached a maximum in summer, a minimum in winter, and an intermediate position during autumn and spring. The concentration of nitrous acid reached a maximum in winter and a minimum in summer. The ratio of nitric nitrogen to nitrous nitrogen was highest in summer and lowest in winter. On many occasions during winter the ratio was approximately as 1 : 1. A relation between atmospheric temperature and this ratio was noted. Its nature was shown by plotting the mean minimum temperature of each month with the mean monthly ratios, the curve being of the same type as those which express changes of chemical velocity with temperature. The ratio is doubled for equal increments of temperature. From the results it would appear that in rain-water nitric and nitrous acids are formed in equal molecular proportions, and that, if the ratio could be determined instantly, or before any change could ensue, it would invariably be as 1 : 1. In cold weather the velocity is retarded to such an extent that little change occurs even after comparatively long periods; hence the increased amounts of nitrous acid found in winter. In hot weather the velocity being greatly increased, the residual amounts of nitrous acid are very small, nearly all having been converted into nitric acid. The facts point to atmospheric nitrogen peroxide as the source of nitric and nitrous acids in rain-water, as this gas reacts with water, forming these acids in equal molecular proportions.

In a graph plotted with daily concentrations of total (nitric plus nitrous) nitrogen as abscissæ, and with rainfall as ordinates, the points are found to arrange themselves into a series of rectangular hyperbolæ. Further, each group of points lying along a particular curve is found to correspond with falls of rain occurring during one particular type of weather. From this it follows that for a particular type of weather (1) the concentration of oxidised nitrogen varies inversely as the rainfall; (2) the product of the concentration and the rainfall is constant; (3) the total weight per unit area of oxidised nitrogen precipitated with rain falling during twenty-four hours is constant. In brief, the amount of oxidised nitrogen per acre carried down by rain falling on any day is a function of the type of weather, and, within certain limits, is independent of the amount of rainfall. These facts may be explained by assuming that for each type of weather there exists in the air a definite concentration of nitrogen peroxide, and that this soluble gas is completely washed out of the air by the first portions of a shower, any further rain falling through the now purified air not increasing the amount of oxidised nitrogen in the rain-water, but, by dilution, decreasing the concentration.

Nine well-defined recurring types of weather have been investigated. These may be classified into three groups, as follows: (1) Antarctic types; (2) tropical types; (3) divided control (Antarctic and tropical) types. The accompanying table shows the number of examples of each type investigated, together with the oxidised nitrogen constant in pounds per 1,000 acres, for each type:

	Number examined.	Oxidised Nitrogen Constant.
Antarctic types:		Pounds per 1,000 Acres.
Λ-shaped Antarctic depressions (*a*) Rear	35	1·5
(*b*) Crest	28	2·5
(*c*) Front	10	4·1
Tropical types:		
Tropical (or monsoonal) depressions (*g*) Spring and autumn type	6	16·0
(*h*) Summer type	3	24·0
(*i*) "Heat-wave" type	2	35·0
Divided control types:		
(*d*) Antarctic depressions with *slight* tropical influence	6	6·1
(*e*) Antarctic depressions with *strong* tropical influence	4	8·5
(*f*) Tropical depressions with *slight* Antarctic influence	5	12·0

Determination of Silver and Base Metal in Precious Metal Bullion.

F. P. Dewey. (*J. Ind. and Eng. Chem.*, 1914, **6**, 650-658, 728-736.)—The determination of silver in bullion, especially in bullion high in gold and low in silver, by the cupellation method, is not as satisfactory as might be desired. The silver, being estimated by the difference between the result of the total fine metal assay and the gold assay, bears the errors of both assays, which may not be compensating, and may be large in proportion to the silver present. Thus, differences of 5 fine are common between the results of two laboratories, and even duplicates may differ by this amount, and that on bullion only 100 to 200 fine in silver. Facts such as these make it desirable that a direct method for silver, applicable to such bullion, should be worked out. Gay Lussac's method has the advantage that, given proper equipment, a large amount of work can be turned out daily; but for accurate work, somewhat elaborate equipment is required, and the method is quite unsuited for occasional work (*cf.* Dewey, Analyst, 1913, **38**, 228). A preliminary difficulty in applying any wet method to bullion of the character referred to is the solution of the silver. The second part of the present paper describes a method which has been, and is still being, investigated in the United States Mints with promising results, though the author expressly states that many details yet require investigation. The principle of the method is alloying the bullion with cadmium, as suggested by Balling (Crookes' "Select Methods of Analysis," 1886, 443), solution of the silver in the alloy by means of nitric acid, and its estimation by Volhard's method.

Some 98 per cent. potassium cyanide is melted in a small porcelain crucible or annealing cup, care being taken that the contents are kept below the boiling-point. The bullion (0·5 grm.), wrapped in about 1 grm. of sheet lead, is immersed in the molten cyanide, and about 5 grms. of stick cadmium immediately added. The alloying operation occupies two to three minutes. When cool, the cyanide is dissolved in water, and the washed button is boiled with 60 c.c. of nitric acid (sp. gr., 32°) until coloured fumes are no longer evolved. The solution is then diluted with 100 c.c. of distilled water, cooled thoroughly (this is important in exact work), and mixed with 5 c.c. of a saturated solution of iron alum. It is then titrated with a solution of ammonium thiocyanate, of which 1 c.c. = 5 mgrms. of silver. In exact work, proofs of approximately the same composition as the samples under examination must be assayed side by side with the samples. G. C. J.

Estimation of Cobalt in High-Speed Steels. L. Dufty. (*J. Iron and Steel Inst.*, 1914, **90**, pp. 14 advance copy.)—The method depends on the precipitation of cobalt as ammonium cobalt phosphate and the ignition of this to cobalt pyrophosphate, a method long in use for the estimation of cobalt in ores. Tungsten is separated first in the usual way, and chromium, molybdenum, and vanadium are separated together with iron by the acetate method. In absence of nickel, the next step is to separate cobalt from manganese, as the latter would also be precipitated by the phosphate reagent. This is readily accomplished by precipitating cobalt as sulphide in weak acetic acid solution, the cobalt sulphide being subsequently redissolved, and cobalt finally precipitated as described below. If nickel be also present, the dimethyl glyoxime method of separation is applied to the original acetate filtrate containing nickel and cobalt, the cobalt being determined by phosphate in the filtrate from nickel after decomposing the excess of dimethyl glyoxime with nitric acid.

The drillings (3 grms.) are dissolved in 50 c.c. hydrochloric acid, and the solution oxidised with the least possible excess of nitric acid. The solution is evaporated to a small bulk, 20 c.c. hydrochloric acid added, and evaporation continued to syrupy consistency. The solution is diluted with about five volumes of 5 per cent. hydrochloric acid, filtered through a paper-pulp filter, and the tungstic acid washed with dilute acid. The filtrate, just short of boiling, is nearly neutralised with ammonia, cooled, diluted to about 500 c.c., and finally ammonium carbonate is added until a slight permanent turbidity results. Twenty c.c. of dilute (1 : 2) acetic acid are added, the mixture boiled, and 25 c.c. of 15 per cent. ammonium acetate solution added. The mixture is boiled for two minutes, transferred to a 750 c.c. flask, made up to 755 c.c. (to allow for volume of precipitate and loss by evaporation during filtering), poured back into the precipitating flask to mix rapidly, and its temperature is taken. It is then filtered through a dry 24 cm. double filter of stout paper, and 500 c.c. collected. Filtered in this way, the temperature of the filtrate is only about 1° C. lower than that of the unfiltered mixture, and the correction (1:1000) is negligible. The 500 c.c. of filtrate (= 2 grms. steel) are treated with 5 c.c. hydrochloric acid and evaporated to about 50 c.c. A slight excess of ammonia is added, and any small precipitate of ferric hydroxide filtered off, washed, and freed from any co-precipitated cobalt by solution in a little hot hydrochloric acid, neutralised and re-precipitated with ammonium acetate. The filtrate is added to the principal solution of cobalt and manganese, and the mixture neutralised with acetic acid, and about 2 per cent. more acetic acid and 15 c.c. of ammonium acetate solution (1 : 2) are added. The mixture is heated to boiling, and hydrogen sulphide passed through it for fifteen minutes. The cobalt sulphide is filtered off on a pulp filter, washed with hydrogen sulphide water containing a little ammonium chloride and acetate, and then dissolved in hot, dilute nitric acid, any small quantity of sulphide undissolved being recovered by igniting the filter and treating the residue with hydrochloric acid and a few drops of nitric acid.

The cobalt solution, now free from manganese, is treated with 20 c.c. hydrochloric acid, evaporated to about 10 c.c., and diluted to about 125 c.c. (50-200 c.c. dilution does not affect the results). Ammonium phosphate (1 grm.) and just enough ammonia to produce a slight precipitate are added, and followed by 15 c.c. of ammonium acetate solution (1 : 2). After heating to the boiling-point, ammonia is added drop by drop until the solution is just faintly alkaline. The beaker is kept in the centre of the hot plate until the bluish flocculent precipitate is transformed into the crystalline purple rose form, a change favoured by the presence of the ammonium salts. The proportions of these ammonium salts may be varied within considerable limits, but smaller amounts retard the transformation to the crystalline form, and very high concentrations (20 grms. ammonium chloride or acetate) double the small amount of cobalt passing into the filtrate. The mixture is further heated for fifteen minutes on the water-bath, with occasional stirring, to insure complete precipitation in the crystalline form, and then filtered on a pulp filter, and the latter washed with hot water. The pulp and precipitate are transferred to a tared platinum dish, dried thoroughly, and then ignited at a full red heat, under which conditions the paper does not char, but burns off rapidly, leaving clean cobalt pyrophosphate. After fifteen minutes in the muffle, the dish is cooled and weighed.

Under the conditions described, the amount of cobalt passing into the filtrate is nearly constant (0·4 – 0·6 mgrm. = 0·02 – 0·03 per cent. on 2 grms. steel), and, for steels containing 1 to 10 per cent. of cobalt is almost exactly balanced by traces of silica and ferric phosphate in the phosphate precipitate. In the most exact work, the cobalt in the filtrate and impurities in the precipitate must be determined, and directions are given for this, but the extreme error involved by neglecting these precautions appears to be 0·02 per cent. on the steel. G. C. J.

Oxygen Content of Open-Hearth Steel. J. A. Pickard and F. M. Potter. (*J. Iron and Steel Inst.*, 1914, **90**, pp. 9, advance copy.)—In a former paper it was suggested that it might be possible to establish some relation between oxygen and carbon content, but the results now published do not support this theory. On the other hand, they supply only one exception to the rule that basic steels are higher in oxygen than acid steels, which are generally admitted to be of better quality than basic open-hearth steels. The highest actual content found even in basic steel (0·042 per cent.) is far lower than figures published by R. H. McMillen (0·12 per cent.; *Metallurgical and Chemical Engineering*, 1913, **40**, 86) and T. Baker (0·081 per cent.; *Carnegie Scholarship Memoirs*, 1909, 219), and it is suggested that the work of these authors was subject to a systematic source of error not recognised.

The method of estimation adopted by the authors is not described in detail, as this has already been done, though not in a journal universally accessible (*Carnegie Scholarship Memoirs*, 1913, 70). It depends on heating the steel (25 grms.) to 1,000° C. in a confined volume of hydrogen with a weighed boat containing phosphorus pentoxide and measuring the increase of weight of the boat. The complete apparatus is obtainable from Müller, Orme, and Co., London, and some additional notes on the method are now added. The most important of these relates to the necessity of rapid transference of the phosphorus pentoxide tube from the weighing tube to the combustion tube, and *vice versa*. How important this is may be realised from the fact that the total increase of weight in an experiment due to water derived from the oxygen in the steel varies from 1 to 10 mgrms., whereas the blank, due to absorption of moisture from the air cannot, by the most skilful manipulation, be kept below 1 mgrm., and may easily become several times as great. By conducting the transference of the boat in a systematic and rapid manner, however, the blank can be kept always between the limits 1 to 1·3 mgrms. G. C. J.

Analysis of High-Grade Metallic Tungsten. H. Arnold. (*Zeitsch. anorg. Chem.*, 1914, **88**, 74-87.)—For this purpose the usual methods for the analysis of tungsten powder are insufficient. Tungsten of over 99 per cent. purity is now a common object of commerce, and is used in so large a proportion in some high-speed steels that the exact determination of the impurities is important. When the total impurities range from 5 per cent. down to 0·2 per cent., large quantities of metal must be taken for the estimation of the minor constituents, and bringing these large quantities of tungsten into solution introduces a preliminary difficulty. Throughout the analysis the tendency of tungsten to form complex compounds is a further source of trouble and greatly limits the choice of methods. The author, having tried many other methods, recommends the following:

Fine tungsten powder is readily enough dissolved by ammonia in presence of hydrogen peroxide, ammonium persulphate, or other suitable oxidising agent. Coarser powder can be treated by the nitrite method, described later, or may be alternately ignited and moistened with nitric acid and then treated with sodium hydroxide solution containing a little hydrogen peroxide, any insoluble residue being reignited and retreated as described. Massive tungsten is most conveniently dissolved electrolytically. It is suspended as the anode in concentrated ammonia contained in a platinum dish which serves as cathode. The addition of 1 to 2 grms. ammonium persulphate for each 5 grms. tungsten to be dissolved is an advantage. With 1 to

2 amps. at 2½ to 3½ volts, the tungsten dissolves at the rate of about 1 grm. in twenty-five minutes. Failing electrolytic appliances, massive tungsten may be fused with alkali nitrite, a method which succeeds still more readily with tungsten powder. The nitrite is heated nearly to its fusion-point, and the tungsten, if massive, is introduced in as small pieces as possible. The reaction being exothermic, the nitrite rapidly melts and the reaction itself is constantly accelerated.

The alkaline or ammoniacal solution is treated with at least 7 c.c. of 50 per cent. tartaric acid solution for each 5 grms. tungstic acid which may be present, diluted considerably (700 c.c. for an original weighing of 20 grms.), and then acidified with hydrochloric acid until about 4 per cent. free acid is present. The tartaric acid keeps the tungsten wholly in solution, whilst the metals of Group 2 are precipitated by passing hydrogen sulphide for several hours through the hot solution, maintaining the gas current during cooling. The solution may set to a gel on standing, but is readily liquefied by warming. The precipitate is filtered off, washed with water free from dissolved oxygen, dissolved in hydrochloric acid containing bromine, and analysed in the usual manner, except that each final precipitate must be tested for silica, and that molybdenum (nearly always one of the chief impurities of commercial tungsten) and arsenic, if present, introduce difficulties. As often as not, arsenic is present in only negligible traces, and it is always worth while to test for it qualitatively, as much needless work may thus be saved. The Gutzeit test serves for this purpose, and at the same time permits an approximate estimation of the amount of arsenic. Suspended tungstic acid does not interfere with the test, which will readily detect 0·01 mgrm. arsenic. In the quantitative analysis, copper, lead, bismuth, and tin having been separated by methods which are described, arsenic, if present, is separated from antimony and molybdenum by taking advantage of the insolubility of arsenic sulphide in strong hydrochloric acid. In the filtrate, antimony and molybdenum are again precipitated as sulphides, which are dissolved in sodium sulphide and antimony determined electrolytically. In the exhausted electrolyte, molybdenum is reprecipitated as sulphide by cautious addition of acid, and finally weighed as MoO_3.

Before proceeding to the estimation of the metals of the ammonium sulphide group in the filtrate from Group 2, the tungstic acid, which may amount to 25 grms. or more, must be separated. This is best effected by pouring the filtrate in a thin stream into a large volume of boiling hydrochloric acid. The bulk of the tungstic acid separates in a dense form, readily washed by decantation with dilute acid. The decanted acid solution is evaporated to dryness and ignited to destroy tartrates, avoiding unnecessarily high temperatures which might give rise to the formation of manganate. The residue contains the oxides or carbonates of the metals to be estimated, together with about 5 per cent. of the tungsten originally present, now in the form of sodium tungstate if sodium salts were used for effecting solution of the sample. If not, it is got into this form and separated from the insoluble oxides and carbonates, after passing carbon dioxide through the mixture to decompose any sodium aluminate. The oxides or carbonates of iron, aluminium, nickel, cobalt, manganese, zinc, calcium, and magnesium are then filtered off and estimated in the usual manner.

Sodium, often present formerly as tungstate in tungsten powder, is wholly absent from modern high-grade tungsten. Cobalt is rarely present, and the proportion of arsenic may be very small, but any of the other metals may be present in a tolerably high-grade product to the extent of 0·2 per cent., which is a notable percentage considering that the highest grades contain less than 0·2 per cent. of foreign metals. The paper does not deal with the non-metallic impurities of commercial tungsten. G. C. J.

Application of Tungsten Salts to the Analysis of Tanning Materials. A. T. Hough. (*J. Soc. Chem. Ind.*, 1914, **33**, 847-848.)—Sodium tungstate gives a precipitate with tannins, the quantitative relationships of which should afford a characteristic "constant" analogous to the "molybdate value" of Lauffmann (*Collegium*, 1913, 10-14). The author employs the tungstate in two forms, alkaline and acid, obtaining different values with the two reagents and the same tannin. It is suggested that the ratio of these two values may afford a means for identifying or controlling the purity of the various tannins, but the author's experiments have been confined to mangrove extracts. The alkaline reagent is prepared as follows: Ten per cent. solution of sodium tungstate, 5 c.c.; 25 per cent. ammonium chloride, 3 c.c.; water, 2 c.c. The acid reagent contains the same quantities of the salts, but with 2 c.c. of 0·85 N-hydrochloric acid instead of the 2 c.c. of water; this gives a liquid which is slightly acid to litmus, but not to methyl orange. Ten c.c. of a filtered solution, containing about 4 grms. of tannin per 250 c.c., are mixed with 10 c.c. of either reagent; the mixture is filtered and 10 c.c. of the filtrate are evaporated to dryness (residue $=A$). The precipitate on the filter is dissolved in hot water, and the solution, together with the washings of the pipette and beaker, is also evaporated to dryness (residue $=B$). The difference between constant dry weights, $B-A$, gives the weight of the precipitate. The percentage of tanning material in 10 c.c. of the extract is then determined from the difference between the total solids and the non-tannin found by the official method (tannin $=C$). Then the ratio

$$\frac{(B-A)\,100}{C}$$

gives the "tungstate value" in the case of the first reagent and the "tungstic value" in the case of the second, and the ratio

$$\frac{\text{tungstic value}}{\text{tungstate value}}$$

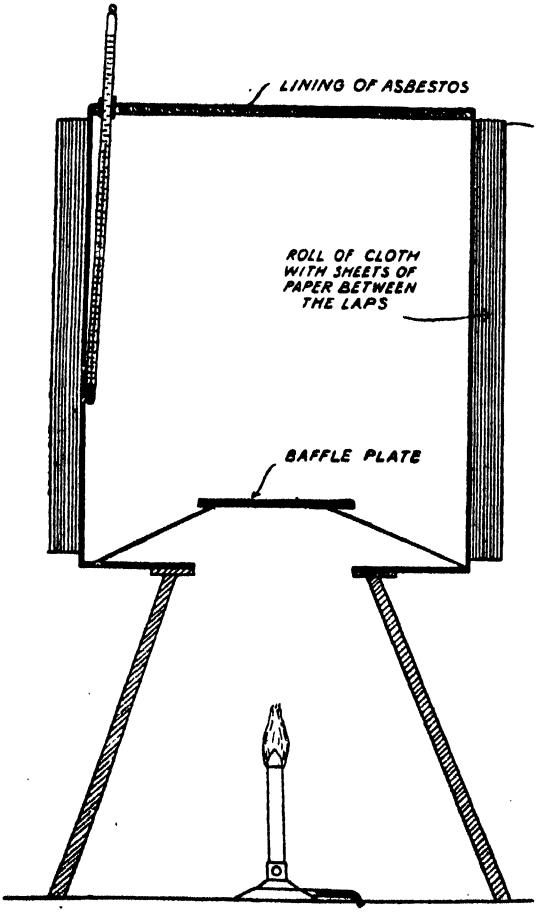
is presumably a constant for any given tannin material. In the case of pure mangrove extract the tungstic value is 156·5 and the tungstate value 149; ratio $=$ 1·0503. J. F. B.

APPARATUS, ETC.

Device to Assist Filtration by Suction. R. A. Gortner. (*J. Amer. Chem. Soc.*, 1914, **36**, 1967.)—In filtering by suction through a Büchner funnel, cracking of a bulky precipitate may be prevented and removal of the mother liquor facilitated by fastening a piece of thin sheet rubber, by means of a rubber band, over the top of the funnel. When the rubber has been drawn down over the top of the precipitate, it may be pressed with a pestle from above without any risk of breaking the filter-paper. Under the pressure thus obtained the precipitate is left nearly dry.

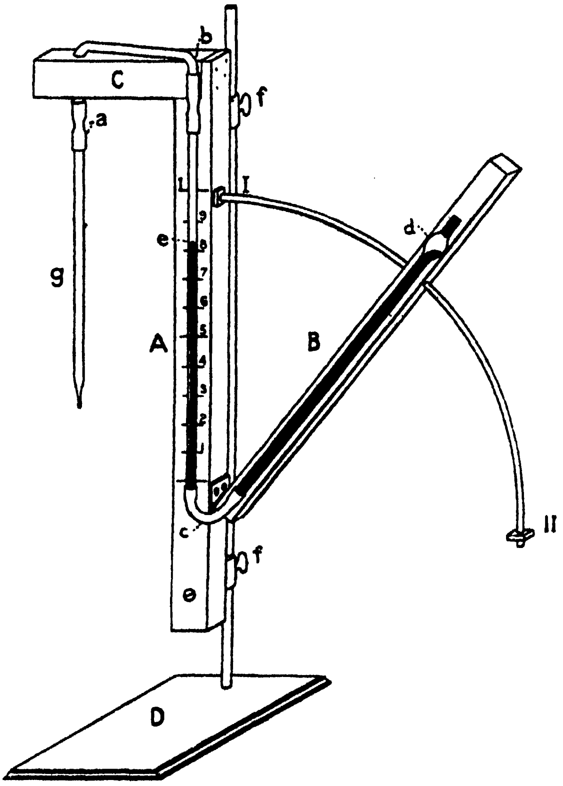
C. A. M.

Apparatus for the Drying of Paper for Test Purposes. C. Beadle and H. P. Stevens. (*J. Soc. Chem. Ind.*, 1914, **33**, 730-731.)—When sheets of paper made on the hand-mould for test purposes are dried loosely without strain, a very considerable contraction takes place, depending on the nature and preparation of the material, and the physical tests are affected by such contraction. The loosely dried paper also becomes cockled, and it is impossible to press the sheets flat. In order to avoid these objections and to obtain sheets more resembling the paper dried on the hot cylinders of the paper-machine, which is held during drying by means of travelling felts, the authors have designed the apparatus illustrated in the figure. It consists of a tin cylinder about 13 inches high and 10 inches in diameter, closed at the top end, and insulated at that part by a plate of asbestos. The bottom end has a hole about 6 inches in diameter, through which the heated air from a Bunsen burner can ascend into the inside of the drum. The hot air from the burner strikes a baffle plate, which deflects it against the walls of the drum, and which is placed in such a position that all portions of the cylinder are uniformly heated. For charging the drying apparatus the drum is placed horizontally on a table and a strip of cloth is tightly wound round it for one and a half revolutions. Sheets of paper are



then placed between the layers of cloth, while continuing to wind the latter round the drum. The cloth is kept as tight as possible, and when the winding is completed, the whole is tied up with tape top and bottom. The cylinder is then placed in position on a tripod with the burner underneath; a flame 2½ to 3 inches long from a ¼-inch burner without air regulator is sufficient to dry fifty small sheets of paper in half an hour. J. F. B.

Pipettometer. W. D. Frost. (*J. Amer. Chem. Soc.*, 1914, **36**, 1785-1787.) —Originally designed for bacteriological work, it is suggested that this instrument may find useful application for other purposes. It consists essentially of the straight glass tubes *g*, *e*, and *B*, and the double right-angled bend *b*, connected by short pieces of rubber tubing as shown. The tube *B* has a bulb, *d*, near the top, and is supported on a wooden arm connected by a hinge to the wooden upright, *A*, to which *e* is fixed. *A* and the rest of the apparatus can be raised or lowered after loosening the set screws, *f f*. With *d* in a vertical position, mercury is poured in until it stands at some convenient high level, such as *I*, in the permanently vertical tube *e*. This point is marked. A beaker of water is brought under the point of the tube *g*, the arm *B* is lowered, the height of the mercury in *e* marked on the wooden backboard, and the contents of *g* discharged by raising *B* and weighed. By repeating this experiment with greater or less lowering of the arm *B*, data for the construction of the scale shown may be obtained. Clearly the interposition of a graduated pipette in place of *e* will not serve the purpose of a scale, as the air between the liquid in *g* and the mercury in *e* is under a variable pressure. G. C. J.



New Seal for Prevention of Aeration in De-aerated Liquids. F. Bachmann. (*J. Ind. and Eng. Chem.*, 1914, **6**, 764-765.)—The apparatus figured in the paper is an ordinary Bunsen valve, with a few pinholes in the rubber instead of the more usual slit. The author had recourse to this appliance for closing bottles filled full of water-sewage mixtures for the methylene blue test for putrescibility. If an ordinary stoppered or corked bottle is used, the stopper is always loosened and some liquid escapes due to expansion of the liquid when placed in the incubator, and on subsequent cooling some air gains entrance. Several appliances have been described for overcoming this difficulty, but none is so simple as or more effective than the Bunsen valve. In the author's view, however, the danger of re-aeration in this test has been somewhat exaggerated, and may be rendered negligible by filling bottles at the incubator temperature and by the use of good corks or stoppers. In a prolonged test, such as the five days' test recommended by the Royal Commission on Sewage Disposal, the use of a Bunsen valve should be valuable. G. C. J.

Freezing-Point of Benzene as a Fixed Point in Thermometry. T. W. Richards and J. W. Shipley. (*J. Amer. Chem. Soc.*, 1914, **36**, 1825-1832.)—The freezing-point of pure benzene is 5·483°±0·002°, on the international hydrogen scale. This fixed temperature may be used, in connection with the freezing-point of water, for calibrating Beckmann thermometers; especial attention must be paid to the temperature of the exposed mercury thread in correcting this interval for use at other temperatures. Benzene, derived from coal-tar or prepared synthetically from benzoic acid, may be purified by shaking it with clean mercury, then with six successive quantities of concentrated sulphuric acid (about two and a half hours each time), followed by washing with water, sodium hydroxide solution, and again with water. The benzene is now shaken for four hours with mercury, then washed with water, dried over calcium chloride and sodium, and distilled. The portion distilling between 80·15° and 80·18° C. (uncorrected) is recrystallised six times and then distilled directly into the Beckmann apparatus in which the freezing-point is determined.

W. P. S.

New Method for Determining the Specific Heats of Liquids. E. J. Hartung. (*British Association Report*, 1914, No. 128.)—The method described, which was suggested by Orme Masson, is a modification of the mixture method for determining specific heats. The principle consists in measuring the lowering in temperature of a known amount of the particular liquid on the introduction of a definite weight of dry ice contained in a thin glass bulb. The calorimeter is a thin glass vessel of about 100 c.c. capacity, and is supported inside a silvered Dewar tube. A well-fitting rubber stopper closes the mouth of this tube, making the apparatus air-tight. Through the stopper is fitted a Beckmann thermometer and also a thin glass stirring-rod, the lower end of which is suitably shaped to receive the small ice-bulb. A third hole, lined with glass and closed with a well-fitting glass stopper, passes through the rubber stopper, and serves for the introduction of the ice-bulb. This last consists of a small sealed, thin glass cylindrical bulb, containing a definite weight of distilled water and also as much silver gauze as possible. The last insures rapid heat conduction, and makes the bulb heavy enough to sink in dense liquids. The bulb is suspended by a fine platinum wire.

The liquid to be experimented with is introduced into the calorimeter by means of a standard pipette, and the apparatus is closed until constant temperature is attained. The ice-bulb has meanwhile been frozen in a mercury bath, supported in an ordinary freezing-point apparatus. When the temperature of the mercury is constant at from − 3° to − 4° C., the bulb is removed by its suspension, and rapidly introduced into the lower part of the stirring-rod in the calorimeter. The liquid is then stirred by hand until constant temperature is again attained, which usually requires about three minutes. Radiation corrections are then applied and the specific heat of the liquid calculated, the heat capacity of the ice-bulb being accurately known. The experiments should be performed in a room regulated to constant temperature.

The advantages claimed for the method are its simplicity, its rapidity, and its accuracy. Experiments with water at 25° C. gave consistent results, agreeing to within 0·4 per cent. When ether was used, it was found necessary to coat the rubber stopper with tinfoil in order to protect it. The results with ether at 25° C. agreed to within 1·4 per cent. (the vapour pressure of ether at this temperature is 545 mm.). The specific heats of several sulphuric-acid-water mixtures were also measured and compared with the classical results of J. Thomsen. The average divergence was less than 1 per cent. Further measurements with different liquids are now in progress.

The apparatus described is not suitable for a viscous liquid (such as glycerol) owing to inefficient stirring. By having another liquid than water in the carrier-bulb, the scope of the method can probably be extended.

New Method for the Determination of Vapour Pressures and an Examination of a Source of Error in Certain Dynamical Methods. F. H. Campbell. (*British Association Report*, 1914, No. 127.)—Since none of the accepted methods is suitable for the determination of the vapour pressure of a binary mixture of a volatile with a non-volatile liquid, a new method has been devised for the purpose.

The principle used is that of allowing a liquid saturated with a suitable gas, usually hydrogen, to evaporate into an enclosed space filled with the same gas at the same temperature and pressure, the latter being approximately atmospheric. The extra pressure exerted by the vapour is measured by means of an open mercury manometer after the volume has been restored to its original value by means of a levelling vessel. The method can be applied to volatile organic liquids of many kinds, since they come into contact with glass and mercury only. The liquid is enclosed in a glass tube projecting through a rubber stopper, which closes an opening at the bottom of the vessel, the stopper being protected from the action of the liquid by a layer of mercury. When the apparatus has reached the temperature of the constant temperature bath in which it is immersed, the tube, previously deeply scratched, is broken by gentle sideways pressure on the projecting end. Saturation of the gas by the vapour is hastened by gently shaking the apparatus.

Experiments have been made with the following liquids and gases: Methyl alcohol, ethyl alcohol, diethyl ether, carbon disulphide, chloroform, and water, evaporating into carbon dioxide, air, hydrogen, and, in the case of chloroform, nitrogen in addition. In every case the values obtained, though very concordant, are lower than the most reliable results obtained by the ordinary static method. The magnitude of the error evidently depends on the solubility of the particular gas in the particular liquid as it diminishes in each case, except that of water, in the order—carbon dioxide, air, hydrogen. With water at 60° C., the results obtained with air and hydrogen are practically identical. The error when hydrogen is used is generally less than 1 per cent., and it is considered that the agreement is sufficient, as with mixtures the ratio between the observed pressure of the solution and that of the pure volatile solvent is to be considered. With air the errors are from 2 to 6 per cent., and it is concluded that methods depending on the saturation of a current of a gas passing through or over solution and pure solvent must therefore involve more or less error from this cause, a fact which does not seem to have been appreciated in the past. Perman (*Proc. Roy. Soc.*, 1903, **72**, 72) and Krauskopf (*J. Phys. Chem.*, 1910, **14**, 489) obtained satisfactory results with water, although those of Regnault (*Ann. Chim. Phys.*, 1845 [3], **15**, 129) and Tammann (*Weid. Ann.*, 1888, **23**, 322), which are lower than those obtained by the static method, are brought into satisfactory agreement with these upon the author's assumption, which indicates that the errors introduced by the presence of a dissolved gas are negligible under certain conditions.

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REPORTS.

Metropolitan Water Board. Eleventh Annual Report for the Year ended March 31, 1914.—The Report consists of twenty-five pages of reading matter and statistics, sixty-six pages of tables and diagrams, and a copious index. The section dealing with the water-supply states that the sources of the supply are the Rivers Thames and Lea, and certain gravel-beds, springs, and wells in Middlesex, Surrey, and Kent. The daily average natural flow of the Thames was 1,418 million gallons, of which the Board abstracted 144 million gallons, or 10·2 per cent., although on occasions of low flow rather more than one-third was abstracted. From the River Lea a daily quantity of 63 million gallons was abstracted out of a flow of 115 million gallons, or 54·8 per cent. The wells and other sources yielded a daily average supply of 41 million gallons. The total daily supply from all sources was 243 million gallons, and the proportions yielded by the various sources were—From the Thames 59 per cent., from the Lea 24 per cent., from wells, etc., 17 per cent. The population supplied was estimated to be nearly 6¾ millions, and the daily supply per head was 36 gallons.

An important part of the Board's work is the storage of the river water before filtration. The capacity of the reservoirs in use for this purpose permits the water from the Thames and Lea to be stored for fifty-two days.

The filtration of the supply is accomplished by 170 acres of sand filters, which provide 0·8 acre of filter surface for each million gallons of water daily. The depth of sand in these filters varies from 2 to 4½ feet, and the rate of filtration through them varied from 0·89 to 1·94 gallon per square foot per hour. Each acre of filtering surface required to be cleaned on the average seven times during the year. The filtered water is pumped to service reservoirs for distribution, of which only the two largest are not covered in.

The results of the chemical and bacteriological examination of the water are summarised very briefly. The number of bacteria in the Thames, Lea, and New River waters, and the effect of the purification on them, were as follow :

	Bacteria per c.c.		
	Gelatine.	Agar.	Bile-Salt.
River Thames	6,178	294	19
,, stored	1,000	50	6
,, filtered	17	3	—
River Lea	11,161	415	31
,, stored	140	11	5
,, filtered	33	4	—
New River	2,372	60	—
,, filtered	15	3	—

Typical *B. coli* were usually found in 0·1 c.c. or 1 c.c. of the Thames and Lea, and in 1 c.c. or 10 c.c. of the New River. After filtration the *B. coli* were usually absent from 100 c.c. of each water.

The chief results of the chemical analysis of the various waters were as follow :

	Parts per 100,000.		Mm. Brown.
	Albuminoid Nitrogen.	Oxygen Absorption.	Colour in 2-Foot Tube.
River Thames	0·0148	0·2076	70
,, stored	0·0128	0·1368	35
,, filtered	0·0054	0·0725	17
River Lea	0·0146	0·1898	71
,, stored	0·0107	0·1128	29
,, filtered	0·0056	0·0754	18
New River	0·0088	0·0891	38
,, filtered	0·0032	0·0390	12

J. H. J.

Metropolitan Water Board. Tenth Research Report. A. C. Houston, June, 1914. The report is divided into seven sections, and continues the work of the author on the detection of the presence of pathogenic organisms in the water-supply of the Metropolis. There are included also criticisms of the work of other authors on cognate subjects.

Search for Pathogenic Organisms in River-Water and Sewage.—In previous reports (ANALYST, 1912, **37**, 195; 1913, **38**, 370) on experiments on river-water it had been found that the typhoid bacillus was absent from 9 c.c. and Gärtner's bacillus from 19 c.c. of the raw water. As the water during filtration is purified about a thousand times, it would follow that the typhoid bacillus must be absent from 9 litres of the water as it is supplied to the consumer. Continuing similar experiments with crude sewage, the author added known numbers of typhoid bacilli to sewage samples from various towns, and was able to isolate them again from 0·01 c.c. of the infected sample. As the numbers of typhoid bacilli added per 0·01 c.c. and those recovered were in the proportion of fifteen to one respectively, it follows that one bacillus would have been detected in one-fifteenth of that amount, namely in 0·00066 c.c. of the sewage, if it had been present. But in a control uninoculated sample of the same sewage, no typhoid bacilli were isolated from 0·01 c.c., although 23,000 colonies were examined, and therefore they must have been absent from 0·00066 c.c. On the average, 704 excremental bacteria are contained in that volume, and also in 17 c.c. of raw river-water, and since the raw water is purified a thousand times, the result is that 17 litres of the filtered water do not contain a single typhoid bacillus. The author considers this conclusion to be reassuring.

Methods for the Isolation of the Typhoid Bacillus.—The author uses a malachite-green solid medium for detecting the presence of typhoid bacilli in sewage. Recently an ox-gall liquid medium has been recommended by the American Public Health Association, and a brilliant green liquid medium by other workers for the isolation of typhoid bacilli from water and fæces. Two series of experiments were made to determine whether there was any superiority in these two enrichment methods over the direct plating method. Samples of sewage were inoculated with large numbers of typhoid bacilli, and portions of 0·01 c.c. examined by each of these methods. In no case were any typhoid bacilli recovered from either of the liquid media, whereas with the solid medium typhoid bacilli were always found.

Influence of Temperature on the Vitality of the Typhoid Bacillus in Water.—In a former report (ANALYST, 1912, **37**, 195) it was shown that the typhoid bacillus survived longer in river-water at a low temperature than at a high temperature. Recently, in America, the reverse statement has been made. The author repeated his experiments, adding typhoid bacilli to river-water and tap-water, and allowing the samples to stand at different temperatures. It was found that at 20° C. the bacilli died out in two weeks, at 10° C. they lived for three to four weeks, and at the freezing-point for three to seven weeks. The point is an important one, as it has a direct bearing on the value of storage in hot and cold climates, and during summer and winter.

Streptococci in Excrement.—This research forms the longest section in the report. It is an attempt to find a reliable distinction between the streptococci of man and of animals. The method employed was to inoculate Conradi-Drigalsky plates with an emulsion of the fæces, and to subculture the minute colonies obtained into litmus lactose broth; those tubes showing acidity were examined microscopically, and, if satisfactory, were subcultured into various sugar media, incubated at 37° C. for three days, and the amount of acidity produced determined with $\frac{N}{20}$ soda. It was found that most human streptococci produced acid in lactose broth, also those from the dog, but very few from the horse, sheep, rabbit, cow, and gull. The amounts of acid produced from dextrose, lactose, and cane-sugar tended to be high in the case of man and low in animals, and from inulin and raffinose, high in the dog and cow, and low in other cases; but the number of intermediate results was so many that no definite differentiation could be drawn. The most important result was that in the case of the horse, sheep, rabbit, and cow, the animals most likely to pollute a water-supply, the excrement was deficient in streptococci, and especially in lactose-positive streptococci, as compared with human excrement. Incidentally it was noticed that the *B. coli* found on the plates from the animal excrement would be classed as identical with those of the human intestine.

Value of Storage.—Objection has been taken to the results obtained by the bacterial counts of the stored water, the criticism being that the diminution in numbers of the bacteria was more apparent than real, the bacteria aggregating into clumps during storage, and each clump being counted as a single bacterium in the plate cultures. To test this point, many series of experiments were made with tap-water inoculated either with *B. coli* or with sewage, and set aside to stand. The bottles were examined in duplicate at intervals during the period of standing, in one case after shaking in the ordinary way, in the other, after adding lead-shot or sand and violently shaking. Almost without exception, no increase in the number of *B. coli* could be found to be brought about by shaking with the shot or sand, which materials might be presumed to break up any clumps of bacilli present. The conclusion appears inevitable either that the disruptive process was not sufficiently violent, or that the diminution of bacteria during storage is due, in fact, to the death of the organisms.

Lime as a Bactericidal Agent.—In America it has been stated that the sterilising action of lime on water is due to the neutralisation of the free and semi-combined carbon dioxide. The author has always found (ANALYST, 1912, **37**, 334) that a slight excess of lime was necessary, and has made further experiments. An emulsion of *B. coli* was added to boiled and unboiled tap-water, and the bacilli were found to survive in each sample for twenty-five days, whereas in a boiled sample to which lime was added they died in seven days. In tap-water exactly neutralised with lime, *B. coli* lived for thirteen days; with small quantities of lime above the neutralisation point they lived over two days, whilst with 6·3 parts of lime in excess they lived for less than one day.

Vitality of the Cholera Vibrio outside the Human Body.—As confirming the author's conclusions with regard to the typhoid bacillus, attention is drawn to a paper by Greig (*Indian J. of Med. Research*, vol. i., No. 3), who finds that uncultivated cholera vibrios die more speedily than cultivated vibrios, and that temperature has a powerful influence on their vitality: in the hot season the life of the organism becomes shorter, whilst in the cold season the maximum duration of life occurs.

J. H. J.

ERRATUM.

Page 439, line 20, *for* "20 parts per million" *read* "10 parts per million.

DECEMBER, 1914. Vol. XXXIX., No. 465.

THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

An ordinary meeting of the Society was held on Wednesday evening, November 4, in the Chemical Society's Rooms, Burlington House. The President, Mr. A. Chaston Chapman, F.I.C., occupied the chair.

The minutes of the previous ordinary meeting were read and confirmed.

Certificates of proposal for election to membership in favour of Messrs. J. K. Crow, D.Sc., F.I.C., and H. Finnemore, B.Sc., F.I.C., were read for the second time; and certificates in favour of Messrs. Harold Stanley Machin, Cleeve, Oatlands Park, Weybridge, analytical chemist with Messrs. Batger and Co.; and Frederic Robinson, M.Sc. (Tech.), A.I.C., The Hollies, Mile End, Stockport, chemist to the executors of F. Robinson, Unicorn Brewery, Stockport, were read for the first time.

Messrs. W. R. Hardwick, B.Sc., F.I.C., H. F. Hills, and R. H. Kay, were elected members of the Society.

The President said that on behalf of the Society a letter had been addressed to the Local Government Board, placing at the disposal of the Government any services that the Society might be able to render in the present crisis; and that letters of acceptance had been received in reply from the Prime Minister and from the War Office.

He asked that the names of all members of the Society who were serving with His Majesty's Forces might be sent to the Honorary Secretaries.

The President also said that members of the Society were invited by the Biochemical Society to attend its meeting to be held at the Lister Institute on Tuesday, December 8, at 5.30 p.m., when papers dealing with a number of processes of what might be called microchemical analysis would be read.

The following papers were read: "Notes on Flour," by R. T. Thomson, F.I.C.; "Methyl Red as an Indicator," by R. T. Thomson, F.I.C.; "Two Rapid Methods of Estimating Water in Crude Petroleum, Oil Fuel, and Similar Substances," by Herbert S. Shrewsbury, F.I.C.; and a "Note on Vinegar," by J. S. Jamieson, F.I.C.

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METHYL RED AS AN INDICATOR.

By R. T. THOMSON.

(*Read at the Meeting, November* 4, 1914.)

In 1908 this indicator was introduced by Rupp and Loose (Analyst, 1909, **34**, 29), who advocated its use because of its delicacy, and especially its value in the titration of ammonia and other comparatively weak bases, such as quinine. H. F. Tizard (*J. Chem. Soc.*, 1910, **97**, 2477) concluded that "as an indicator methyl red is greatly superior to methyl orange, as the colour curves show. The end-point is very much sharper than with methyl orange. Methyl red is, in fact, an extremely sensitive indicator, and should come into extensive use." However valuable this investigation may be in its special line, such sweeping conclusions on the data collected are very apt to be misleading to the analyst, and as a matter of fact they have been so. It is quite true that the end-point with methyl red is sharper than with methyl orange when these indicators are simply tested in water with acid or alkali. The difference is greater in solutions of neutral salts, such as chloride and sodium sulphate, in which case the sensitiveness of methyl red is not much affected, whereas that of methyl orange, as I pointed out in 1883, is considerably lessened. So far there is agreement, but the subsequent portions of the quotation given would naturally lead one to suppose that methyl red could be substituted for methyl orange, and naturally it has been concluded that it would act similarly to the latter in every particular, but with greater delicacy. Finding, as stated by Rupp and Loose, that methyl red was much superior to methyl orange for the titration of ammonia, a few tests were made to determine its value for other substances. The following figures show the percentages of alkali or acid, calculated on 100 parts of the actual substance tested, when methyl red was employed as indicator, the titration being made in the cold with standard sulphuric acid and sodium hydroxide respectively.

	Per Cent. found.
Carbonate of soda	about 90·0
Boric acid	„ 0·2
Acetic acid	„ 94·0
Lactic acid	98·0

The end-point of the reaction in the case of lactic acid was fairly good, but the others, and especially sodium carbonate, showed such gradual change in colour that the result was of no value. The same is the case with sulphurous, phosphoric and tartaric acids. If quinine be dissolved in a slight excess of sulphuric acid and titrated with sodium hydroxide, using methyl orange as indicator, the end-point is about where the normal sulphate is produced, but the change in colour is obscure. With methyl red this occurs also when the normal sulphate is formed, the end-point being good in a hot solution, which is required to keep the salt in solution. The compound referred to is the quinine sulphate of the British Pharmacopœia, which is also neutral to litmus.

The above result with sodium carbonate is very low owing to the influence of the carbonic acid which is liberated, but if the solution is boiled off after each addition an excellent end-reaction is obtained. As free carbonic acid has practically no influence on methyl orange, it is apparent that methyl red belongs to a different category, and is, in fact, similar to litmus. This difference and similarity are also observable in the case of various acids, as well as the quinine, referred to above, so the assumption that methyl red can be used as a substitute for methyl orange has no foundation in fact. As a general rule it may be taken to resemble litmus in its indications, but rare instances may occur, as has been the case with other indicators, in which it may give a different result. It is thus imperative that, before any indicator is used for a special purpose, its general similarity to another indicator should not be depended upon, but its property in the particular circumstances should be carefully tested. In one of my previous papers (*J. Soc. Chem. Ind.*, 1887, 6, 195) I divided indicators into three classes: (1) The methyl orange group, (2) the litmus group, and (3) the phenolphthaleïn group; but I should now place methyl red as the one most resembling litmus, since, on the whole, including the more decisive change in colour and smallness of quantity of material required, it is to be preferred to the latter.

DISCUSSION.

Mr. F. W. F. ARNAUD said that he had found methyl red to be very useful, taking the place, in most instances, of methyl orange. While it could not be used in the presence of large quantities of carbonic acid, it was perfectly easy to titrate solutions of sodium carbonate with acid, using methyl red, provided that the solution was kept practically at boiling-point. It was a particularly useful indicator at night, as the colour-change was then very marked and easily discernible, whereas in the case of methyl orange the colour-change was sometimes difficult to see by artificial light. Methyl red was a very good indicator for use in the Kjeldahl process.

Mr. E. HINKS said that he used methyl red exclusively in the Kjeldahl process, but not much for other purposes, because of its liability to failure through the presence of carbonic acid.

* * * * *

NOTES ON FLOUR: 1. ACIDITY OF FLOUR. 2. NATURAL AND ARTIFICIAL BLEACHING OF FLOUR. 3. SULPHATES AND LIME IN FLOUR.

BY R. T. THOMSON.

(*Read at the Meeting, November* 4, 1914.)

EARLY in 1912 I was engaged in a case, under the Food and Drugs Acts, in connection with bleached flour, and in this paper I propose to put on record some of the results of experiments made at that time, and since then.

1. "ACIDITY" OF FLOUR.

In Jago's "Technology of Bread-Making," and in other works, it is taken for granted that flour is acid, and in the work named (1911 edition, p. 773) the use of phenolphthaleïn as indicator is recommended, and in calculating the result the instruction is in the following terms: "Assuming that the acidity of meal or flour is due to lactic acid (as undoubtedly it is in whole or great part)," etc. In Wood's 'The Story of a Loaf of Bread" (1913, p. 68) it is stated that "wheat, like almost all plant substances, is slightly acid"; and these statements appear to be the last words on the subject at present. The last-named book does not mention the indicator employed, although the former does; but both are inexact or careless in the use of terms which should be defined with accuracy as to what is actually meant. Acidity and alkalinity are words which may mean anything according to the indicator employed, an apposite example of which is fresh milk, which is acid to phenolphthaleïn, alkaline to methyl orange, and amphoteric or practically neutral to litmus. Accordingly a bald statement could be made with apparent accuracy that milk is acid, alkaline, or neutral, in conformity with the point of view taken. In flour we have in certain respects a mixture similar to milk, as it contains proteins, carbohydrates, and phosphates, so that an unqualified assertion of acidity is unscientific. In Jago's volume, besides phenolphthaleïn, there is mention of turmeric as an indicator for acid in flour; but I showed many years since that these two substances behave similarly and belong to the same class, so that there is no corroboration of acidity. In the same volume Planchon is referred to as having made various tests and to have come to the conclusion that the proper way to test the "acidity" of flour is to suspend 5 grms. in water and titrate with standard caustic soda, using phenolphthaleïn as indicator. Now, there is no valid reason for coming to such a conclusion, as is quite apparent from the results obtained with different indicators. In order to test this point, methyl orange, methyl red, and phenolphthaleïn, were selected as representing most clearly the three classes of indicators in general use.

A first-grade flour from wheat grown in Scotland was made the subject of experiment, and the titrations were made in three different solutions prepared from 5 grms. of the sample. In the first case 10 grms. were suspended in 400 c.c. of water, and 200 c.c. filtered off after standing for one hour; in the second 5 grms. were suspended in 200 c.c. of water for one hour; and in the third 5 grms. were heated to boiling with 200 c.c. of water, and allowed to cool for one hour. These solutions were then titrated, with the following results:

	Acidity by Phenolphthaleïn. SO_3 per Cent.	Reaction to Methyl Red.	Alkalinity by Methyl Orange. NaOH per Cent.
Filtered sample	0·056	neutral	0·042
Unfiltered sample	0·114	neutral	0·108
Boiled sample	0·114	neutral	0·108

The reason for adopting SO_3 and NaOH as expressing acidity and alkalinity is that SO_3 (80) is equal to 2NaOH (80), so that the figures are interchangeable.

These results are somewhat startling, and give no support to the assumption that flour is actually acid. It may be argued that phenolphthaleïn, being most sensitive to acids, is the proper indicator to use; but in a complicated mixture like flour there might be some disturbing element which is apparently acid, whereas, like sulphate of ammonia, it may be really neutral. The mineral matter of the flour was treated with hot water, filtered, and titrated with $\frac{N}{10}$ hydrochloric acid. The ash insoluble in water was dissolved in a measured quantity of the same acid, and titrated back with $\frac{N}{10}$ caustic soda. In each case the results were calculated on the same basis as already described, and the results were as follows:

	Per Cent.
Water	12·31
Ash soluble in water	0·29
Ash insoluble in water	0·08
Total mineral matter	0·37
Alkalinity of soluble ash to methyl orange	0·042
Alkalinity of insoluble ash to methyl orange	0·038
Alkalinity of total mineral matter	0·080
Alkalinity of soluble ash to phenolphthaleïn	neutral
Alkalinity of insoluble ash to phenolphthaleïn	0·012

These results account to a large extent for the alkalinity of the original flour to methyl orange, but there is still a little, the presence of which cannot be explained satisfactorily. It may be due to the apparent alkaline reaction of some organic ingredient, but it must also be borne in mind that the end-point of the reaction with flour is obscure, and not reliable within narrow limits. As regards phenolphthaleïn, the mineral matter as a whole is slightly alkaline, and the soluble portion is neutral, which indicates that the ash consists largely of mono-acid potassium phosphate (K_2HPO_4), or rather the corresponding pyrophosphate. Of course, this does not help to explain the apparent acidity of the flour when phenolphthaleïn is used as an indicator.

In addition to the above, twenty samples of flour, the sources of which were wheat from Scotland, America, Canada, Russia, etc., were examined, and all were found to be alkaline to methyl orange, practically neutral to methyl red and litmus, but acid to phenolphthaleïn. The acidity by the last-named indicator varied from 0·096 to 0·136 per cent. of SO_3. It is against all reason to suppose that this apparent acidity is due to the presence of free lactic acid, according to Jago's confident statement, as it has been well known for a long period that lactic acid can be estimated with fair accuracy if litmus, especially in the form of paper, is used as an indicator. In my paper on methyl red I have shown that fully 98 per cent. of lactic acid can be determined by that indicator, so that it is obvious that free lactic acid cannot exist in flour when it is practically neutral to both litmus and methyl red. In order to corroborate this, flour was extracted with ether and alcohol respectively, the solution evaporated after mixing with a little water, then filtered and titrated, when only minute traces of acid were found. As the average acidity of flour is equal to about 0·25 per cent. of lactic acid, we have now absolute proof that flour does not contain that acid in the free condition, and, indeed, that proportion would make flour quite sour to the taste. So far the proof of non-acidity is negative, and I am not in a position as yet to state positively what the apparent acidity to phenolphthaleïn is due to.

When flour becomes "sour" it shows acidity to methyl red and litmus, and this increases in that condition to phenolphthaleïn, whereas the alkalinity to methyl orange is not reduced. This is apparent from the following results obtained with a sound flour, and one, of similar composition, which had become sour:

	Per Cent.	
	Sound.	Sour.
Water	12·44	13·10
Mineral matter	0·36	0·35
Alkalinity of filtered solution (methyl orange)	0·048	0·054
Acidity of filtered solution (methyl red)	neutral	0·160
„ „ „ (phenolphthaleïn)	0·060	0·312
Alkalinity when suspended in water (methyl orange)	0·110	0·080
Acidity „ „ „ (methyl red)	neutral	0·294
„ „ „ „ (phenolphthaleïn)	0·112	0·455

It is only necessary to note that, in spite of the production of a considerable proportion of acid, methyl orange still indicates alkalinity; but methyl red shows a substantial amount of acid, especially when the flour is suspended in water during titration. It is generally supposed that lactic acid is produced by the souring of flour, but this is doubtful, because, when extracted with alcohol or ether, only a very small proportion of the additional acid was found. Further experiments will be necessary in order to determine what acid or acids have been produced during the passage of sound flour into the sour condition.

Although no definite conclusion has been reached as to what the apparent acidity of flour is due, there can be no doubt that it is not actually acid, except perhaps to a minute extent, and the statement that acidity is undoubtedly due to lactic acid is a mere theoretical fallacy. From the results recorded, it is evident that any appreciable acidity due to the souring of flour can be detected and determined by titration with standard caustic soda, using methyl red solution or litmus-paper as indicator.

2. Natural and Artificial Bleaching of Flour.

The discussion on this subject hinges on the theories (1) that natural bleaching by exposure to the atmosphere is similar to artificial bleaching by nitrogen peroxide, and (2) that these two processes are essentially different. In dealing with this question, it will be necessary to know the composition of air as regards nitrites, and to trace the production of flour from the grain to the final bleaching. In order to do this, I visited at a flour-mill, and observed the process of milling, procuring samples of the wheat and the different products for subsequent examination.

The first point, however, was to test the air for nitrites, several plans for this being tried; but the best was the following adaptation of published methods: A thoroughly cleaned stoppered bottle capable of holding about 4 litres was procured, filled with air in the mill by drawing it in with a pump, then 100 c.c. of water containing 2 c.c. of the Griess-Ilosvay reagent were poured into the bottle, and the latter closed. The bottle was now turned about occasionally, so that the liquid spread over the sides, and finally the contents were left overnight. Three or four stoppered bottles, capable of holding rather more than 100 c.c., containing different quantities of standard sodium nitrite in 100 c.c. water, and 2 c.c. of the Griess-Ilosvay reagent, were also left overnight, and then compared with the solution from the air, when, by a comparison of the colours, it was possible to determine the amount of nitrite present. The following are the results obtained in the air of the mill referred to:

	Mgrm. in 10 Litres.	Grain per 1,000 Cubic Feet.
Nitrous anhydride (N_2O_3)	0·0061	0·267
Equal to sodium nitrite	0·0110	0·484

Samples of the wheat, and products of the milling of it, were taken and tested, with the following results:

	Nitrites as Sodium Nitrite.	Colour by Lovibond's Tintometer.	
	Part per Million.	Yellow.	Red.
Wheat	0·16		
First-grade flour ...	0·51	0·65	0·32
Second-grade flour ...	0·30	0·84	0·40
Fine thirds	0·18		
Common thirds	0·16		
Bran	0·20		
Germ	0·12		

It will be apparent that the first-grade flour has absorbed 0·35 part per million of nitrites from the atmosphere, and for this to take place 1 pound of flour would have to take up all the nitrites contained in 5 cubic feet of air, and 1 ton would require 11,200 cubic feet. This result is not at all surprising, as the flour falls through the atmosphere in a fine dust, which would pick up most of the nitrites in the air. As the product becomes coarser, the absorption of nitrites naturally becomes less. Of unbleached flour from various sources, the nitrites ranged from 0·10 to 0·62 part per million.

We have next to consider the cause and effect of natural bleaching, or exposure of flour to the influence of the atmosphere under different circumstances, as compared with artificial bleaching with nitrogen peroxide.

A sample of unbleached flour was selected and tested for nitrites, and 24 ounces of it were placed in a thin, slightly glazed paper bag, and kept in a light, well-ventilated room for thirty-four days. At the end of this period ½ ounce was carefully removed from the surface, and this was called "first layer." Another sample of ½ ounce was taken from the flour immediately below where the top layer had been, and this was named "second layer." One ounce of the flour in direct proximity to the sides of the bag was next taken, and the remaining 22 ounces were thoroughly mixed, and the four samples thus drawn were analysed separately. In addition, a portion of the remaining 22 ounces was bleached with 5 c.c. of nitrogen peroxide gas per kgrm. of flour, and two quantities of 2 ounces each were exposed to a certain atmosphere for half an hour and two hours respectively, in open Petri dishes 6 inches in diameter, having thus an exposed surface of about 28 square inches. The atmosphere referred to was that of an almost hermetically closed safe, about 600 cubic feet in capacity, and in which two jets of gas were burning, with a consumption between them of about 6 cubic feet per hour. The results of the tests were as follows:

	Nitrites.	Yellow Colour.	Red Colour.	Total Colour destroyed. Per Cent.
Original sample before exposure ...	0·34	0·86	0·36	—
First layer	3·12	0·80	0·34	6
Second layer	2·50	0·80	0·34	6
From sides of bag	2·90	0·80	0·34	6
Remaining 22 ounces	0·58	0·86	0·36	0
Average analysis	0·83	0·85	0·35	2
After half-hour's exposure in air	3·05	0·80	0·34	6
After two hours' exposure in air ...	6·20	0·76	0·32	11
After bleaching with 5 c.c. NO_2 per kgrm.	3·06	0·64	0·28	24

Water ... ... 14·74 per cent.
Mineral matter ... ... 0·42 „

The nitrites are stated as sodium nitrite, parts per million.
The colour was taken in Lovibond's tintometer.

It will be observed that, in the first layer of the flour contained in the bag, the nitrites have risen from 0·34 to 3·12 parts per million, that on the sides of the bag to 2·90 parts, while the second layer is 0·62 less than the first layer, showing that penetration by the atmosphere is slow, or at least that the nitrites in it are absorbed by a thin layer of the flour. Taking this fall in nitrites as being regular into the centre of the flour, it was found, by taking the measurement of the bag into account, that the influence of the atmosphere as indicated by nitrites had ceased when it had penetrated ½ inch into the flour, the bag being 4 inches in height, 4½ inches in breadth, and 2½ inches in thickness. From these considerations it was apparent that three-fourths of the flour had been totally unaffected, and in any event the whole sample, had it been mixed together, would only have contained 0·83 part of nitrites per million. Under comparatively unfavourable conditions, therefore, the absorption of nitrites from the air is very small, and it was found that a fairly well glazed bag prevented even such absorption for months.

If we now make a comparison of the analysis of the first layer of flour, that of the portion exposed for half an hour in the special atmosphere referred to, and that of the portion bleached with 5 c.c. of NO_2 gas per kgrm., we find that the proportion of nitrites is practically the same in each. The percentage of colour destroyed, however, is entirely different in each case, that by exposure in the bag in the top layer being only 6 per cent., the same being the case with the flour exposed for half an hour, whereas by artificial bleaching no less than 24 per cent. has been removed. The same thing can be shown by another example of portions of the same flour being exposed in Petri dishes as before, in different localities, for ten days, the following being the results obtained:

	Nitrites.	Colour. Yellow.	Colour. Red.	Colour destroyed. Yellow.	Colour destroyed. Red.	Colour destroyed. Total.
Original sample	0·42	0·64	0·30	—	—	—
Pure smokeless air ...	1·30	0·42	0·26	34	13	27
Medium smoky air ...	3·32	0·38	0·24	37	20	34
Smoky air	4·50	0·38	0·24	37	20	34

None of this flour was available for bleaching, but another flour bleached with 2 c.c. of NO_2 per grm., showed 1·40 parts of nitrites per million, but only 7 per cent. of colour removed, showing clearly that the destruction of 27 per cent. in the pure smokeless air, and only an absorption of 1·30 of nitrites, is due to other causes than nitrous acid, probably chiefly oxidation, assisted by moisture and light. Those who approve of the principle of tampering with flour by bleaching with nitrogen peroxide very ingeniously draw an analogy between this and the natural bleaching, such as has been described, and hold that they are identical, only that the one process is rapid and the other slow. In face of the results given, it is evident that this is a mere theory, with no data whatever on which to build the top-heavy edifice, but, while the argument so far is convincing, it can be clinched by other data which explain the reason why so much nitrite is present without having the bleaching effect that might be expected.

It is generally assumed that when flour is bleached by nitrogen peroxide, a mixture of nitric and nitrous acids is first produced, but it is very doubtful whether this reaction occurs before bleaching is effected. The colouring matter of flour does not appear to be intimately associated with any of the constituents, and as bleaching with nitrogen peroxide is practically instantaneous, it is probable that it acts to a large extent as such, and not after the acids have been formed.

The various flours mentioned above were tested by mixing with water, sodium nitrite, potassium iodide, and a little starch solution (the latter being scarcely necessary, but added for safety), then allowed to stand for several hours, and in some cases for twenty-four hours. In no case was iodine liberated, which means that no trace even of free nitrous acid was present, as this test is extremely delicate, because there is continuous regeneration of nitrous acid from the nitric oxide at first formed, and the blue colour becomes gradually stronger owing to the accumulative action taking place. It was found that a flour having an apparent acidity of 0·114 per cent. of SO_3 to phenolphthaleïn required the addition of lactic or hydrochloric acid equivalent to 0·08 per cent. of SO_3 before any trace of iodine was liberated. The acid required to bring the flour into a condition capable of liberating nitrous acid is practically equivalent to the alkalinity of the mineral matter to methyl orange—that is, to the point at which the di-acid phosphate (KH_2PO_4 or other analogous compound) is formed. It was found by experiment that KH_2PO_4 in solution liberated nitrous acid almost immediately, but a mixture of 80 per cent. of that compound with 20 per cent. of K_2HPO_4 had no action even after twenty-four hours. The flour in question had an alkalinity of 0·108 per cent. to methyl orange, so that rather more acid is required than four-fifths of the alkalinity of the ash (0·08 per cent.), but not the full amount indicated by methyl orange. Some flours require rather less acid than this, and others rather more, before free nitrous acid would be produced, but acidity equal to fully 700 parts of nitrous acid per 1,000,000 parts of flour, would be required to be absorbed by flour before any of that acid would be in the free condition. Even admitting that atmospheric air is slightly acid, which I have never found it to be, enormous volumes would have to come in contact with flour before there could be any liberation of nitrous acid. As nitrites do not bleach flour, we have reduced to absurdity the plausible theory that nitrous acid is the active agent in bleaching flour exposed to the atmosphere. Since reaching these conclusions Dr. G. W. Monier-Williams published his report (Food Reports, No 19) in which he proves the difference between natural and artificial bleaching, but on different lines from those I have followed. It is therefore apparent that the supporters of bleaching flour have not proved that the compounds produced are not deleterious, nor have their opponents proved the opposite view. I may note here that L. Weil (ANALYST, 1909, **34**, 102) stated that sulphuretted hydrogen gas when passed over bleached flour brought back the original colour. I made some experiments in this direction, leaving the flour in contact with sulphuretted hydrogen for several hours, and once under a sealed bell jar overnight, but the colour had evidently been destroyed, there being no restoration visible.

3. SULPHATES AND LIME IN FLOUR.

The determination of these is important where sulphates are to be allowed for in self-raising flour, or in testing whether a flour contains persulphate of potash. The method of analysis I have found most suitable is to mix 20 grms. of the flour with 250 c.c. of water containing about 15 c.c. of hydrochloric acid of about 1·16 sp. gr., heat in a vessel of boiling water until the gelatinous starch becomes liquid, and then boil gently for a few minutes. Now allow the solution to cool, filter off the bulky precipitate, chiefly proteins, wash with dilute hydrochloric acid of the same strength as the sample was dissolved in, heat to boiling, and add a solution containing 1 grm. of barium chloride. Allow to stand for at least twelve hours, collect the precipitate on a double Swedish filter, wash carefully and thoroughly with cold water, dry, ignite, and weigh. If this process be carried out exactly as described, there will be no difficulty in the filtration, which proceeds rapidly and gives clear solutions. The following are results obtained in some of the products of the wheat in which nitrites have already been given:

	Per Cent.			
	First-Grade Flour.	Second-Grade Flour.	Fine Thirds.	Common Thirds.
SO_3	0·011	0·023	0·048	0·061
CaO	0·015	0·034	0·060	0·102
Mineral Matter ..	0·400	0·820	2·240	

Other samples of flour containing from 0·35 to 0·42 of mineral matter showed from 0·010 to 0·013 per cent. of SO_3. These are even higher than the results found by Cripps and Wright (ANALYST, 1914, 429), but our methods of testing differ, and I have not had an opportunity of comparing their process with mine. The sulphates evidently rise and fall with the ash, and the ratio is practically the same for first-grade and second-grade flour, but not for thirds. It would appear that a fair allowance for SO_3 naturally present in a flour would be one-fortieth of the mineral matter, if no other mineral matter has been introduced.

DISCUSSION.

The PRESIDENT said that, whilst he had personally no experience in regard to the acidity of wheaten flour, he had had a good deal in connection with that of barley flour and malt, and he should have thought that at the present day the bulk of the acidity of these grain infusions, when fresh, would never be attributed to lactic acid. It had been shown years ago by Fernbach, who was one of the first to investigate the matter, that the greater part of the acidity of these infusions was due to various acid phosphates, and this was confirmed later by Matthews and others. The phosphates most likely to be present were KH_2PO_4 (neutral to methyl orange and acid to phenolphthaleïn) and K_2HPO_4 (alkaline to methyl orange and neutral to phenolphthaleïn); and a liquid containing a mixture of these two phosphates would clearly be acid to phenolphthaleïn on account of the one and alkaline to methyl orange on account of the other. The fact that in flour which had become sour the alkalinity to methyl orange remained about the same as in the original flour would seem on the face of it to indicate that the organic acids formed were incapable of converting the one phosphate into the other; but there might be other and more probable explanations. The production on ignition of a mixture of pyrophosphate and metaphosphate, the former alkaline and the latter acid to phenolphthaleïn, would suffice to explain the results obtained by the author in his examination of the ash of the flour. The whole matter was rendered more complicated by the probability of the presence in small quantities of other phosphates—*e.g.*, those of magnesium and calcium. For a long time it was the custom, in examining malt, to return the acidity as lactic acid, but this had been almost entirely abandoned for a good many years. Litmus and certain other indicators were useless in this connection, because they did not yield definite indications with the phosphates referred to. As Mr. Thomson had said, the best indicators for the purpose were methyl orange and phenolphthaleïn. With regard to the question of bleaching, he should like to point out what he considered to be the inadvisability, if it could possibly be avoided, of expressing Lovibond tintometer values in terms of mixed series. It was impossible to make a valid comparison of results expressed in more than one series, because, for instance, 6 yellow + 2 red was not the same as 5 yellow + 3 red. It was better, in his opinion, to try and make an approximate match with a single series whenever

Mr. C. A. SEYLER thought that the increase in acidity on souring must be due originally to formation of an organic acid. The change which took place was probably due to bacterial action, which must result in decomposition of organic matter, and therefore one would expect some organic acid to be formed, lactic acid being the most probable. Lactic acid was a far more powerful acid than KH_2PO_4, its dissociation constant being 1380×10^{-7}, as against only 2×10^{-7} for KH_2PO_4. If lactic acid was formed, it would be capable of converting K_2HPO_4 into KH_2PO_4, and would then disappear in the form of potassium lactate, which was alkaline to methyl orange; so that the alkalinity to methyl orange would remain about the same; but the acidity to phenolphthaleïn would be increased in proportion to the lactic acid formed.

The PRESIDENT said that he did not suggest that organic acids were not formed; but it was an undoubted fact that, in the case of malt flour at any rate, the acidity was largely due to these phosphates.

Dr. MONIER-WILLIAMS said that the most important point in Mr. Thomson's observations as to bleaching was the very small penetration of nitrous acid or nitrites into flour when it was kept in an atmosphere containing the products of combustion; and the fact that, whereas artificial bleaching resulted in the presence of nitrite together with the destruction of colour, the absorption of nitrite from the air resulted merely in the presence of nitrite without destruction of colour. He very much doubted whether it was possible to estimate accurately amounts of nitrite less than 1 part per million, since in order to obtain, with such minute traces, colours which are at all comparable, the standard nitrite solutions must be prepared with the aqueous extract from a sample of unbleached flour containing no trace of nitrites; and in practice, this is difficult of attainment. He thought that in proceedings relating to bleached flour, the question of the deleterious action of nitrites themselves had been given rather too much prominence, because it was open to the defence to say that the actual quantity of nitrite present was too minute to have any physiological effect. It was probable that the alterations produced in the body of the flour itself—such as the destruction of the colouring matter—might be of more importance than the presence of minute amounts of nitrite. The colouring matter of flour seemed to be identical with carotin, a highly unsaturated hydrocarbon, which probably played an important part in plant physiology in certain directions connected with plant respiration. Willstätter has shown that the colouring matters of flour, yolk of egg, green leaves, and also of the *corpus luteum* of the animal body, were closely allied, and were presumably derived from the same substance. Until more was known on the subject it seemed dangerous to say that the destruction of even a small proportion of such a constituent in a foodstuff was without any effect on the properties of the foodstuff itself. With regard to nitrates, of course if flour containing moisture came in contact with nitrous fumes, nitrates as well as nitrites would be formed, but he thought it might be assumed that nitrates or nitric acid would not affect the colouring matter, which was probably in solution in the oil of the flour. What probably happened was that the gas was absorbed to a small extent by the oil of the flour. It had been shown that in order to produce the elaidin reaction a certain minimum proportion of nitric oxide must be absorbed by the oil, forming an additive compound first, after which the elaidin reaction proceeded. It seemed very likely that such an additive compound was formed when the gas came in contact with the oil of the flour, and that the absorbed nitric oxide combined with the colouring matter, quite apart from any subsidiary reactions due to interaction of the excess of gas with the moisture in the flour. It was interesting to note that practically all flour contained some proportion of nitrites. In the case of untreated flour, ammonium nitrite might be absorbed from the air, and there was also the possibility of the production of nitrites from nitrates by bacterial or enzymic action. On the other hand, if bleached flour were kept for a long time, the nitrite content tended towards a certain minimum—about 1 part per million; so that, whether in bleached or in unbleached flour, a balance seemed to be struck in regard to the proportion of nitrite present after the flour had been kept for some time.

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TWO RAPID METHODS OF ESTIMATING WATER IN CRUDE PETROLEUM, OIL-FUEL, AND SIMILAR SUBSTANCES.

By HERBERT S. SHREWSBURY, F.I.C.

(*Read at the Meeting, November* 4, 1914.)

The author has devised the following methods in view of the fact that those generally in use are tedious, lengthy, and not very accurate.

I. Distillation Process.—The only novelty in the method is the apparatus devised, which it is hoped will be found simple and effective.

A 500 c.c. distillation-flask is supported with its side-tube at right angles to the bench and inserted to the neck in a 25 c.c. cylinder, graduated in tenths or, better, in twentieths, with a bore not much greater than that of the side-tube. The cylinder is supported in a vessel provided with an outlet at the bottom, through which water is passed at such a rate as to keep the level constant within a few millimetres of the top. Such an arrangement forms an efficient combination of condenser and receiver, and practically eliminates any loss of water.

One hundred c.c. of oil (or a volume equivalent to 100 grms. calculated from the sp. gr.) are placed in the flask with a few pieces of dry pumice. The neck and surface of the flask (uncovered by the oil) are now heated by a Bunsen flame, the flame being kept in continual movement playing over the surface of the glass. In this way these portions of the flask (commencing from the neck close to the cork) are kept at such a temperature that no water can condense inside the flask; the heating is continued from above downwards, with occasional heating below the oil surface.

By careful manipulation it is possible to drive off all the water, and the process is finished by distilling several c.c. of the oil itself. No drops of water should be allowed to condense and roll down into the hot oil, as this is liable to cause violent bumping. If necessary, on account of the percentage of water present, a larger receiver may be used, or it may be changed for others of the same size. It is of course necessary to keep the side-tube of the flask above the level of the distillate.

The distillate is finally rapidly rotated, to assist the settling of the water, which may be further accelerated by the addition of dry petroleum ether. When no further increase in its volume is observed, the percentage of water is read.

II. TURBIDITY TEMPERATURE PROCESS.—This method depends on: (1) The possibility of extracting water from oil-fuel with glacial acetic acid; (2) the delicacy with which the turbidity temperature of glacial acetic acid and a standard oil responds to minute percentages of water.

The method has been found to agree well with the distillation process. The main disadvantages are the preparation of a standard oil and the necessity of making careful standards for every new stock of glacial acetic acid. The turbidity temperatures do not vary with increase of water as regularly as one would wish, but by making a table of standards in tenths per cent. of water from 0—2·6 per cent., it is possible to obtain a water percentage very near to the truth. The irregularity of the figures may be due to the difficulty of determining the exact turbidity-point. It is the author's practice to note the temperature at which the mixture becomes suddenly and markedly turbid. This point is slightly below the temperature of first faint turbidity. A suitable standard oil (giving a turbidity temperature of 49° C. with glacial acetic acid) was made by mixing equal volumes of arachis and coconut oils, the latter of which is truly an oil at tropical temperatures.

A correction is necessary for the effect on the turbidity temperature of substances other than water dissolved from the oil fuel. This is made by obtaining a blank on the oil previously dried by boiling for a short time in an open dish. When the standards are prepared, an estimation may be made in less than ten minutes.

Description of Method.—Ten c.c. of standardised glacial acetic acid are placed in a dry, stoppered, graduated 25 c.c. cylinder and 10 c.c. of the oil-fuel are added. The cylinder is shaken and its contents transferred to a dry separator. The acetic acid extract is separated and passed through a dry filter, and 2 c.c. of the filtrate are heated with 2 c.c. of the standard oil in a test-tube until the mixture is clear. (The heating is conveniently effected by a spirit lamp.)

A thermometer is inserted in the clear fluid and the turbidity temperature observed in the usual way. After subtracting the blank, which is obtained by similarly treating 10 c.c. of the dried oil and subtracting from the turbidity temperature obtained that of the standard acetic acid, reference to the standards gives a direct reading of the weight of oil in 100 volumes of the oil, and the figure may be calculated to a percentage by use of the sp. gr. of the oil. With oils of a sufficiently similar nature, a constant blank may be used. When the percentage of water is above 2·5, it is necessary to dilute the acetic acid extract with standard glacial acetic acid, exactly the same dilution being made of the extract which gives the figure for the blank. In these cases, after subtracting the blank, the water percentage indicated by the table is multiplied by the ratio of dilution.

Turbidity Temperature of Standards.—The following table shows some turbidity temperatures of the oil mixture mentioned and some standards prepared from a sample of glacial acetic acid. It is, of course, necessary for each analyst to fix his own standards, and these would have to be freshly prepared for every new stock of glacial acetic acid.

Acetic Acid Standard. Per Cent. of Water added.	Turbidity Temperature. ° C.
Nil	49
0·5	63
1·0	74
1·1	75
1·2	76
1·3	79
1·4	82
1·5	86
1·6	88
1·7	90
2·0	93

It will be seen that the average increase in turbidity temperature is 2·2° C. per tenth per cent. of water.

An extract of dried oil fuel made with the same glacial acetic acid in the way described gave a turbidity temperature of 57·5° C., furnishing a blank of 8·5° C.

Comparison of Methods.—The following comparative results were obtained by the two methods for various samples of crude petroleum :

Sample.	Percentage of Water.	
	Distillation Method.	Turbidity Temperature Method.
A	0·00	0·00
B	0·23	0·23
C	1·00	1·15
D	1·40	1·45
E	2·65	2·58
Preparation containing 1 per cent. water	1·00	1·30
Preparation containing 10 per cent. water	9·70	10·00

Application of Methods to Other Substances.—Both methods are probably applicable (with modifications unessential in principle) to the estimation of water in butter, margarine, lard, and other oils and fats, and generally to substances immiscible with water and capable of distillation, or substances from which water may be removed by extraction with glacial acetic acid.

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Discussion.

Mr. G. N. Huntly said that he had used a direct distillation method somewhat similar to, but rather more elaborate than, the one now described. The distillation was carried out in a current of air, the main fraction being collected in freezing mixture and the rest in drying tubes. This was an accurate method, but tedious. Another method which was said to be a good one was the toluene method. About ½ litre of the oil (the quantity depending upon the proportion of water expected) was distilled with about 100 c.c. of toluene, the distillate being collected in a graduated tube. When checked by the addition of known quantities of water to dry oil, this method was shown to be accurate practically to within 0·01 per cent., depending on the graduation of the tube. It was not quite so good as the direct distillation method for crude oil, because in that case the meniscus did not form well and was difficult to read accurately.

Dr. Lessing said that from a comparison of the figures, it would seem very much simpler to use the distillation method than the turbidity method, which latter seemed rather complicated and necessitated constant preparation of fresh standards. In the distillation method, however, when the water was present only in minute traces—less than 0·1 per cent.—the distilled oil absorbed almost as much water as was separated; and the same applied to the toluene method or any method in which a lighter oil was used. These methods had been used a good deal not only for oil but for coal; and the necessity had been shown by Constam and by Marcusson, and was confirmed by experiments which he himself had made, that the toluene or xylene, or whatever light oil was used, should have a definite moisture content. It was best to distil the oil with water before each determination, so that it assumed a certain degree of moisture, and did not absorb any of the moisture that was driven off. Attention was drawn to Sanders' method, recently described in the *Journal of the Chemical Society.*

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Notes on Alcoholometry. II. A. F. Joseph and W. N. Rae. (*J. Soc. Chem. Ind.*, 1914, **33**, 991-992.) (*Cf.* Analyst, 1913, **38**, 508.)—Further notes on the relative merits of Bedford's tables and the ordinary ones. The former are preferred, on the whole, both at high and low temperatures. The errors or inconsistencies of Bedford's tables rarely exceed the error which would be caused by an error of 1 division in the reading of a hydrometer. G. C. J.

Method by which the Corrected Sikes's Tables in Use in India, for determining the Proof Strengths of Spirits at Temperatures from 40° F. to 100° F. by Sikes's Hydrometer, were calculated. F. G. Carter. (*J. Soc. Chem. Ind.*, 1914, **33**, 993-994.)—The author describes in detail how these tables were calculated, starting with the tables in the Analyst (1880, **5**, 42), making use of the formulæ for expansion of Baumhauer, Recknagel, Kopp, and others, data derived from these formulæ being tested by direct experiment. G. C. J.

Ethyl Ester of Linolic Tetrabromide as a Product in the Analysis of Cottonseed Oil. L. S. Palmer and P. A. Wright. (*J. Ind. and Eng. Chem.*, 1914, **6**, 822-823.)—On brominating the unsaturated fatty acids of cottonseed oil (obtained by extracting the lead soaps with ether and decomposing the ether-soluble portion with hydrochloric acid) the authors obtained, in place of the expected linolic tetrabromide, a crystalline compound, m.-p. 58° C. This compound on analysis was found to consist of ethyl-bromo-linolate, $C_{17}H_{31}Br_4COOC_2H_5$. The cause of its formation has not yet been ascertained, but it appears that the changes which result in its production take place when the lead soaps are in contact with ether.

W. P. S.

Index List of Trade Names of Synthetic Drugs, New Remedies, Photographic Developers, etc. Compiled chiefly from "The Extra Pharmacopœia," W. H. Martindale and W. W. Westcott, fifteenth edition, 1912; "Die Arzneimittel-Synthese," S. Fränkel, III Aufl., 1912; "Synthetic Drugs or Medicinal Products," V. Coblentz, in Thorpe's "Dictionary of Applied Chemistry," 1913, vol. v., 357-380; and "Merck's Index," III Aufl., 1910. (Through *J. Soc. Chem. Ind.*, 1914, 33.)—The following is a supplement to the list formerly published (*J. Soc. Chem. Ind.*, 1900, **19**, 1131):

Abanone.—Magnesium phosphotartrate.

Acetomorphine.—Diacetylmorphine, $C_{17}H_{17}NO_3(C_2H_3O)_2$.

Acetopyrin.—Antipyrine acetylsalicylate, $C_{11}H_{12}N_2O,C_2H_3O.OC_6H_4.COOH$.

Acetosal.—Acetylsalicylic acid, $C_2H_3O.OC_6H_4.COOH$.

Acetozone.—Mixture of equal parts of acetyl-benzoyl peroxide, $C_6H_5CO.O.O.C_2H_3O$ and kieselguhr.

Acetysal.—See Acetosal.

Acidol.—Betaine hydrochloride, $C_5H_{11}NO_2,HCl$.

Acitrin.—2-Phenylquinoline-4-carboxylic acid ethyl ester, $C_9H_5N(C_6H_5)CO_2C_2H_5$.

Acopyrin.—See Acetopyrin.

Adalin.—Bromodiethylactylurea $(C_2H_5)_2CBr.CO.NH.CO.NH_2$.

Adamon.—Dibromodihydrocinnamic acid bornyl ester.

Adigan.—Digitalis preparation containing all the active principles except digitonin, this having been removed by precipitation with cholesterol in alcoholic solution.

Adrenal.—Crystalline base from the suprarenal gland.

Adrenaline.—*o*-Dihydroxyphenylmethylaminomethylcarbinol hydrochloride, $(HO)_2C_6H_3.CH(OH).CH_2.NH(CH_3),HCl$.

Adreucaine.—See Eudrenine.

Afridol.—Sodium salt of hydroxymercury-*o*-toluic acid, $CH_3.C_6H_3(CO_2Na)HgOH$.

Agnin.—Adeps lanæ or purified wool fat.

Agnolin.—See Agnin.

Agobilin.—Strontium cholate mixed with small quantities of strontium salicylate and diacetylphenolphthaleïn.

Agurin.—Addition product of theobromine-sodium and sodium acetate, $C_7H_7N_4O_2Na,C_2H_3O_2Na$.

Albargin.—Gelatose-silver (15 per cent. Ag).

Aldoform.—A formaldehyde preparation.

Aleudrin.—Carbamic acid ester *αα*-dichloroisopropyl alcohol, $(CH_2Cl)_2.CH.O.CO.NH_2$.

Alformin.—Sixteen per cent. solution of basic aluminium formate, $Al_2(OH)_2(HCO_2)_4$.

Alginoid Iron.—See Algiron.

Algiron.—Iron compound of alginic acid (from seaweed); contains 11 per cent. Fe.

Allmatein.—Condensation product of hæmatoxylin and formaldehyde, $CH_2O_2(C_{16}H_{12}O_5)_2 : CH_2$.

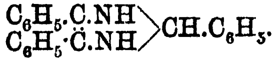
Allosan.—Santalyl allophanate, $NH_2.CO.NH.COO.C_{15}H_{23}$.

Alphogen.—Succinyl peroxide $(COOH.CH_2.CH_2.CO)_2O_2$.

Alphozone.—See Alphogen.

Althein.—Asparagin, $C_2H_3(HN_2)(COOH)CO.NH_2 + H_2O$.

Alypin.—Benzoyl-1·3-tetramethyldiaminoethyl-2-isopropyl alcohol hydrochloride, $C_6H_5CO.O(C_2H_5)C[CH_2.N(CH_3)_2]_2,HCl$.

Amarin.—Triphenyldihydroglyoxaline, 
$\begin{matrix} C_6H_5.C.NH \\ C_6H_5.C.NH \end{matrix}\!\!>\!CH.C_6H_5$.

Amenyl.—Methylhydrastimide hydrochloride, $CH_8O_2.C_6H_2(CO)_2N(CH_3),HCl$.

Amidopyrin.—Dimethylaminoantipyrine, $C_{11}H_{11}N_2O.N(CH_3)_2$.

Amphotropin.—Hexamethylenetetramine camphorate, $(C_6H_{12}N_4)_2,C_8H_{14}(COOH)_2$.

Amylarine.—Isoamyltrimethylammonium hydroxide, $C_5H_{11}(CH_3)_3N.OH$.

Anæsthesin.—Ethyl ester of *p*-aminobenzoic acid, $NH_2.C_6H_4.CO_2C_2H_5$.

Anæsthone.—See Anæsthesin.

Analutos.—See Kalmopyrin.

Angioneurosin.—Nitroglycerin, $C_3H_5(O.NO_2)_3$.

Anisotheobromine.—Addition product of theobrominesodium and sodium anisate, $C_7H_7N_4O_2Na,C_6H_4(OCH_3)CO_2Na$.

Anogon.—Mercury salt of 2·6 di-iodophenol-4-sulphonic acid, $C_6H_2I_2(OHg)(SO_3Hg)$.

Antacedin.—Calcium saccharate.

Anthrasol.—Coal tar distillate having colour and consistence of olive oil.

Antiarthrin.—Condensation product of saligenin and tannin, probably the hydroxybenzyl ester of tannic acid.

Antileprol.—Purified chaulmoogra oil.

Antiluctin.—Potassium-ammonium antimonyltartrate, $[SbO(C_4H_4O_6)_2KNH_4]H_2O$.

Antiperiostin.—Mercury iodocantharidate, $C_8H_{11}IO(COO)_2Hg$.

Antipyoninum.—"Neutral" sodium tetraborate, prepared by fusing together borax and boric acid.

Antisclerosine.—Mixed inorganic salts of normal blood.

Antodin.—Phenyl ether of glycerol, $C_3H_5(OH)_2O.C_6H_5$.

Aperitol.—Mixture of equal parts of isovaleryl- and acetyl-phenolphthaleïn.

Aponal.—Amyl carbamate, $NH_2.COOC_5H_{11}$.

Appallagin.—Mercury compound of nosophen (tetraiodophenolphthaleïn).

Apyron.—Lithium acetyl salicylate, $C_2H_3O.OC_6H_4.COOLi$.

Archibromin.—Monobromoisovalerylglycolylurea, $CH_2(OH).CO.NH.CO.NH.CO.C_4H_8Br$.

Archiodin.—Monoiodoisovalerylglycolylurea, $CH_2(OH).CO.NH.CO.NH.CO.C_4H_8I$.

Argaldin.—Combination of albumin-silver and hexamethylenetetramine (10 per cent. Ag).

Argoferment.—Colloidal silver.

Argyrol.—Silver-vitellin (20 to 30 per cent. Ag).

Arhéol.—Santalol, $C_{15}H_{24}O$.

Arhoin.—Addition product of diphenylamine and ethyl-thymyl-benzoate,
$(C_6H_5)_2NH,C_{10}H_{13}.C_6H_4.CO_2C_2H_5$.

Aristochin.—See Aristoquinine.

Aristoquinine.—Carbonic ester of quinine, $CO(OC_{20}H_{23}N_2O)_2$.

Arrhenal.—Sodium methylarsinate, $AsO(CH_3)(ONa)_2,6H_2O$.

Arsacetin.—Sodium *p*-acetylaminophenylarsinate,
$C_2H_3O.NH.C_6H_4AsO(ONa)(OH),3—4H_2O$.

Arsacetin-Quinine.—Contains 43 per cent. of arsacetin and 54 per cent. of quinine.

Arsalyt.—Bismethylaminotetra-aminoarsenobenzene.

Arsamin.—See Atoxyl.

Arsan.—Glidin preparation containing silver.

Arsanilic Acid.—*p*-Aminophenylarsinic acid, $NH_2.C_6H_4AsO(OH)_2$.

Arsen-phenol-amine.—See Salvarsan.

Arterenol.—*o*-Dihydroxyphenylaminomethylcarbinol hydrochloride,
$(HO)_2C_6H_3.CH(OH).CH_2NH_2,HCl$.

Asiphyl.—See Asyphil.

Aspirin, Soluble.—See Kalinopyrin.

Aspirophen.—Aminoacetphenetidide acetylsalicylate,
$C_2H_5O.C_6H_4.NH.C_2H_2O.NH_2,C_2H_3O.OC_6H_4.COOH$.

Asquirrol.—Mercury dimethoxide, $(CH_3O)_2Hg$.

Astrolin.—Antipyrine methyl-ethyl-glycollate.

Asurol.—Addition product of mercury salicylate and sodium aminohydroxyisobutyrate.

Asyphil.—Mercury salt of atoxyl, $(NH_2C_6H_4.AsO.OHO)_2Hg$.

Atophan.—2-Phenylquinoline-4-carboxylic acid, $C_9H_5N(C_6H_5)COOH$.

Atoxyl.—Sodium *p*-aminophenylarsinate, $NH_2.C_6H_4.AsO(OH)(ONa),xH_2O$.

Atrinal.—Atropine sulphuric acid, $C_{17}H_{25}O_6NS$.

Atyrosyl.—See Asyphil.

Azovernin.—Acetylaminoazotoluene, $CH_3.C_6H_4.N_2.C_6H_3(CH_3).NH.C_2H_3O$.

Barbitone.—See Veronal.

Barutin.—Addition product of theobromine-barium and sodium salicylate.

Benzinoform.—Carbon tetrachloride.

Benzosalin.—Methyl benzoyl salicylate, $C_6H_4(O.CO.C_6H_5)COOCH_3$.

Biogen.—Magnesium peroxide.

Biolactyl.—Preparation of lactic acid bacilli.

Bisciniod.—Combination of cinchonidine hydriodide and bismuth iodide,
$C_{19}H_{22}N_2O,HI,BiI_3$.

Bismon.—Colloidal bismuth.

Bismutose.—Contains about 22 per cent. Bi and 66 per cent. albumin.

Blankit.—Sodium hydrosulphite.

Blenal.—Carbonic acid ester of santalol, $CO(OC_{15}H_{23})_2$.

Bornyval.—Bornyl isovalerate, $C_4H_9CO.O.C_{10}H_{17}$.

Bornyval, New.—Bornyl isovaleryl glycollate, $C_4H_9COOCH_2.COOC_{10}H_{17}$.

Boroform.—Solution of formaldehyde in sodium glyceroborate.

Borovertin.—Hexamethylenetetramine triborate, $C_6H_{12}N_4,3HBO_2$.

Borsalyl.—Sodium borosalicylate.

Bromeigon.—Albumin preparation containing bromine.

Bromelia, Cryst.—β-Naphthyl ethyl ether, $C_{10}H_7.O.C_2H_5$.

Brometone.—Tribromo tertiary butyl alcohol, $CBr_3.C(CH_3)_2OH$.

Brominol.—Brominated sesamé oil (33 per cent. Br).

Brominoleum.—See Brominol.

Bromochinol.—Acid dibromosalicylate of quinine, $C_{20}H_{24}N_2O_2,2C_6H_2Br_2(OH)COOH$.

Bromoglidin.—Wheat gluten preparation containing 10 per cent. Br.

Bromogluten.—Vegetable albumin preparation containing 8 per cent. Br.

Bromolein.—Brominated unsaturated fatty acids of almond oil (20 per cent. Br).

Bromoprotein.—See Bromogluten.

Bromotan.—Bromotannin-methylene-urea.

Bromural.—α-Bromoisovalerylurea, $C_4H_8Br.CO.NH.CO.NH_2$.

Brophenin.—Bromoisovalerylphenocoll; a brominated phenetidine derivative.

Brovalol.—Bornyl bromovalerate, $C_4H_8Br.COO.C_{10}H_{17}$.

Bynin.—Liquid malt extract.

Cacodyliagol.—Guaiacol cacodylate, $(CH_3)_2AsO.O.C_6H_4(OCH_3),H_2O$.

Cajuputol.—Cineol (eucalyptol), $C_{10}H_{18}O$.

Calcidine.—Calcium iodide.

Calcinol.—Calcium iodate.

Calcusol.—Effervescent preparation of potassium bicarbonate and piperidine-*p*-sulphamine benzoate, $SO_2(NH_2)C_6H_4COOH.C_5H_{11}N$.

Calomelol.—Colloidal calomel.

Camphoid.—Collodion substitute; 1 part of pyroxylin in 20 parts each of camphor and absolute alcohol, by weight.

Camphoral.—Camphoric acid ester of santalol, $C_8H_{14}(COO)_2(C_{15}H_{23})_2$.

Camphosan.—Solution of 15 parts of camphoric acid methyl ester in 85 parts of santalol.

Canadol.—Light petroleum spirit, sp. gr. 0·650 to 0·700.

Casumen.—Soluble form of casein ("flocculent casein"); contains 90 per cent. of protein.

Celloidin.—Pyroxylin purified by solution in alcohol and ether.

Cellotropin.—Benzoic ester of arbutin, $C_6H_4OC_6H_{11}O_5.O.COC_6H_5$.

Ceredine.—Special dry powdered yeast.

Cerolin.—Solid extract of yeast, containing fat.

Cetiacol.—Pyrocatechol methyl cetyl ether, $C_6H_4(OCH_3)(OC_{16}H_{33})$.

Chinaphenin.—See Quinaphenin.

Chinaphthol.—See Quinaphthol.

Chineonal.—See Quineonal.

Chininum lygosinatum.—See Quinine lygosinate.

Chinoform.—See Quinoform.

Chinosol.—See Quinosol.

Chloralamid.—Chloral formamide, $CCl_3.CH(OH).CO.NH_2$.

Chloretone.—Trichloro tertiary butyl alcohol, $CCl_3.(CH_3)_2C.OH,\frac{1}{2}H_2O$.

Chlorin.—Dinitroresorcinol, $(NOH)O : C_6H_2 : O(NOH)$.

Chlorobrom.—A solution of potassium bromide 6, chloral formamide 6, and water 58 parts.

Chloryl Anæsthetic.—Ethyl chloride.

Chromoform.—Combination of methyl hexamethylenetetramine and dichromic acid, $(C_6H_{12}N_4CH_3)_2Cr_2O_7$.

Chrysoform.—Dibromo-diiodohexamethylenetetramine, $C_6H_8Br_2I_2N_4$.

Cibrola.—Preparation of milk and glycerophosphates.

Cicatricine.—A solution containing thiosinamine 20, antipyrine 33, and eucaine lactate 0·65 part per 100.

Citarin.—Sodium anhydromethylenecitrate,

$O\left\langle\begin{matrix}CH_2\\CO\end{matrix}\right\rangle CO\left\langle\begin{matrix}CH_2.COONa\\CH_2.COONa\end{matrix}\right.$.

Citrocoll.—Neutral aminophenacetin citrate.

Cocæthylin.—Ethyl ester of benzoylecgonine, $C_6H_5CO.C_8H_{13}NO.COOC_2H_5$.

Codeonal.—Mixture of 1 part of medinal and 2 of codeine-veronal.

Colalin.—Cholalic acid, $C_{24}H_{40}O_5,H_2O$.

Coryfin.—Menthyl ethyl glycollate, $CH_2O(C_{10}H_{19}).COOC_2H_5$.

Coxpyrin.—See Acetosal.

Cresegol.—Mercury-potassium salt of cresolsulphonic acid.

Cresosteril.—Acid ortho-oxalic ester of *m*-cresol,

$CH_3.C_6H_4O.C(OH)_2.C(OH)_2.OC_6H_4(CH_3)$.

Cryogenin.—*m*-Benzaminosemicarbazide, $NH_2.CO.6_6H_4,NH.NH.CO.NH_2$.

Cupferron.—Ammonium compound of nitrosophenylhydroxylamine.

Cupratin.—Copper albuminate.

Cupri-aseptol.—Copper *m*-phenol-sulphonate, $(HO.C_6H_4.SO_3)_2Cu$.

Cupricin.—Cuprous cyanide.

Cuprocitrol.—Copper citrate, $2Cu_2C_6H_4O_7,5H_2O$.

Cuprol.—Compound of nucleinic acid and copper.

Cusylol.—Soluble copper citrate.

Cycloform.—Isobutyl ester of *p*-aminobenzoic acid, $C_6H_4(NH_2)COOC_4H_9$.

Cypridol.—Mercuric iodide (1 per cent.) in sterilised oil.

Cystamin.—See Hexamine.

Cystogen.—See Hexamine.

Cystopurin.—Addition product of hexamethylene tetramine and sodium acetate,

$C_6H_{12}N_4,2C_2H_3O_2Na,6H_2O$.

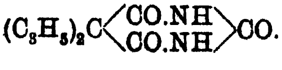
Deba.—See Veronal.

Dentalone.—Solution of chloretone in essential oils.

Dermogen.—See Ektogen.

Desalgin.—"Colloidal chloroform"; an albuminous substance combined with about 25 per cent. $CHCl_3$.

Dial-Ciba.—Diallylbarbituric acid,



$(C_3H_5)_2C\left\langle\begin{matrix}CO.NH\\CO.NH\end{matrix}\right\rangle CO$.

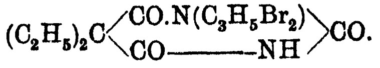
Diaspirin.—Succinylsalicylic acid, $(CH_2.COO)_2(C_6H_4COOH)_2$.

Digipotene.—Preparation containing the whole of the glucosides of digitalis leaves.

Dioform.—Acetylene dichloride, CHCl : CHCl.

Diogen.—Sodium salt of aminonaphtholdisulphonic acid, $NH_2.C_{10}H_4(OH)(SO_3Na)_2$.

Diogenal.—Dibromopropyldiethylbarbituric acid,



$$(C_2H_5)_2C\left<\begin{matrix}CO.N(C_3H_5Br_2)\\CO\text{———}NH\end{matrix}\right>CO.$$

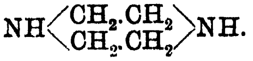
Dioxogen.—A 3 per cent. solution of hydrogen peroxide.

Diplosal.—Salicylosalicylic acid, $HO.C_6H_4.CO.O.C_6H_4.COOH$.

Dipropæsin.—Urea derivative containing the residues of 2 mols. of propœsin, $CO(NH.C_6H_4.CO_2C_3H_7)_2$.

Disalol.—Phenyl salicylosalicylate, $HO.C_6H_4.CO.O.C_6H_4COOC_6H_5$.

Dispermin.—Piperazine,



$$NH\left<\begin{matrix}CH_2.CH_2\\CH_2.CH_2\end{matrix}\right>NH.$$

Durine.—A formalin preparation.

Dymal.—Didymium salicylate.

Edinol.—*m*-Amino-*o*-hydroxybenzyl alcohol hydrochloride, $HO.C_6H_3(NH_2)CH_2OH,HCl$.

Ehrlich-Hata "606."—See Salvarsan.

Ektogen.—Zinc peroxide.

Elarson.—Strontium salt of chloroarsenobehenolic acid (13 per cent. As).

Electrargol.—Electrolytic colloidal silver.

Embarin.—Sodium mercurisalicyl sulphonate.

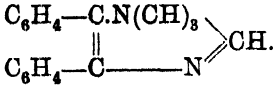
Empyroform.—Condensation product of tar and formaldehyde.

Enesol.—Mercury salicylarsenate (38 per cent. Hg).

Epinene.—3.4-Dihydroxyphenylethylmethylamine, $(HO)_2C_6H_3.CH_2.CH_2.NH.CH_3$.

Epinephrine.—*o*-Dihydroxy-4-methylaminoethanolbenzene, $(HO)_2.C_6H_3.CH(OH).CH_2.NH.CH_3$.

Epiosine.—1-Methyl-4·5-diphenyleneimidazole,



$$\begin{matrix}C_6H_4\text{—}C.N(CH)_3\\ \|\\ C_6H_4\text{—}C\text{———}N\end{matrix}\Big>CH.$$

Epirenan.—See Adrenaline.

Erepton.—Meat preparation in which proteins are completely resolved into amino-acids.

Ericin.—See Mesotan.

Ervasin.—Acetyl cresotinic acid.

Escalin.—Aluminium powder mixed to a paste with glycerol.

Estoral.—Menthyl borate, $B(O.C_{10}H_{19})_3$.

Euboruyl.—*a*-Bromoisovaleric ester of borneol, $C_4H_8Br.COOC_{10}H_{17}$.

Eucodeine.—Codeine methyl bromide, $C_{18}H_{21}NO_3,CH_3Br$.

Eucol.—Guaiacol acetate, $CH_3O.C_6H_4O.C_2H_3O$.

Eudrenine.—A solution containing 0·01 grm. of eucaine and 0·03 mgrm. of adrenaline per c.c.

Euguform.—Acetylated methylenediguaiacol.

Eulatin.—Mixture of *p*-bromobenzoic acid, *o*-aminobenzoic acid, and antipyrine.

Eumydrine.—Methylatropine nitrate, $C_{16}H_{20}NO_3(CH_3)_2NO_3$.

Euphyllin.—Mixture of equal proportions of primary and secondary theophylline ethylene diamine, $C_2H_4(NH_2)_2,C_7H_8N_4O_2 + C_2H_4(NH_2)_2,2C_7H_8N_4O_2$.

Eupnine.—Caffeine preparation containing iodine.

Euporphine.—Apomorphine methyl bromide, $C_{17}H_{17}NO_2.CH_3Br,H_2O$.

Eupyrine.—*p*-Phenetidine-vanillin ethyl carbonate,
$C_6H_4(OC_2H_5)N : CH.C_6H_3(OCH_3)O.CO_2C_2H_5$.

Euquinine.—Quinine ethyl carbonate, $C_2H_5O.CO.OC_{20}H_{23}N_2O$.

Euresol.—Resorcinol monoacetate, $C_6H_4,(OH)(O.C_2H_3O)$.

Euscopol.—Inactive scopolamine hydrobromide, $C_{17}H_{21}NO_4,HBr$.

Eustenin.—Addition product of theobromine-sodium and sodium iodide,
$C_7H_7N_4O_2,NaI$.

Exodin.—Mixture of diacetylrufigallic acid tetramethyl ether 23, rufigallic hexamethyl ether 30, and acetylrufigallic pentamethyl ether 47 per cent.

Fæxin.—See Ceredine.

Fermenlactyl.—Preparation of lactic acid bacilli.

Feronuclin.—Dry yeast extract.

Ferratogen.—See Ferrinol.

Ferrichthol.—Ichthyol preparation containing iron.

Ferrinol.—Iron nucleinate; contains 21 per cent. Fe_2O_3 and 2½ per cent. P.

Fibrolysin.—Fifteen per cent. solution of a combination of thiosinamine (2 mols.) and sodium salicylate (1 mol.).

Fluoram.—Ammonium bifluoride.

Forgenin.—Tetramethyl-ammonium formate, $H.COON(CH_3)_4$.

Formamint.—Preparation obtained from 1 mol. of lactose and at least 5 mols. of formaldehyde.

Forman.—Chloromethyl menthyl ether, $C_{10}H_{19}O.CH_2Cl$.

Formicin.—Formaldehyde-acetamide, $CH_3.CO.NH.CH_2OH$,
or $CH_3.C(:NH).O.CH_2OH$.

Formidin.—Methylenedisalicylic iodide, $C_{15}H_{16}O_6I_2$.

Formitrol.—Formaldehyde preparation.

Formolyptol.—See Formitrol.

Formurol.—Addition product of Hexamethylene tetramine and sodium citrate,
$C_6H_{12}N_4,C_6H_7O_7Na$.

Fredo.—Calcium hydrosulphite.

Furfurin.—A glyoxaline derivative, $C_3H_3N_2(C_4H_3O)_3$.

Gaduol.—Extract containing the alcohol-soluble constituents of cod-liver oil.

Gallogen.—Ellagic acid, $C_{14}H_6O_8$.

Gastrosan.—Bismuth bisalicylate.

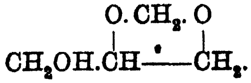
Gaultheriasalol.—Methyl salicylosalicylate, $HO.C_6H_4.CO.OC_6H_4.COOCH_3$.

Glidine.—A protein preparation from wheat.

Glonoin.—Nitroglycerine.

Glycarbin.—Glyceryl carbonate.

Glycerin-formal.—A condensation product of formaldehyde and glycerol,



$$\begin{array}{r} O.CH_2.O \\ CH_2OH.CH\text{——}CH_2. \end{array}$$

Glycobrom.—Glyceryl ester of dibromohydrocinnamic acid, $C_3H_5[O.CO.(CHBr)_2C_6H_5]_3$.

Glycocyamine.—Guanidine acetic acid.

Glycosal.—Monosalicylic ester of glycerol, $C_3H_5(OH)_2.O.CO.C_6H_4(OH)$.

Gomaine.—Solution of iodoform and camphor in sesamé oil.

Griserin Novum.—Iodohydroxyquinoline sulphonic acid, $C_9H_4I(OH)(SO_3H)N$, mixed with 20 per cent. $NaHCO_3$.

Guaiachinol.—Quinine dibromoguaiacolate, $C_6H_2Br_2(OCH_3)OH,C_{20}H_{24}N_2O_2$.

Guaiamaltin.—Mixture of malt extract and potassium sulphoguaiacolate.

Guatannin.—Guaiacol tannocinnamate.

Gynoval.—Isobornyl isovalerate, $C_4H_9.COOC_{10}H_{17}$.

Hectine.—Sodium benzosulpho-*p*-aminophenylarsinate, $C_6H_5.SO_2.NH.C_6H_4.AsO(OH)(ONa)$.

Hegonon.—Ammonio-silver nitrate-albumose (70 per cent. Ag).

Helcosol.—Basic bismuth pyrogallate, $HO.C_6H_3O_2BiOH$.

Helicon.—See Acetosal.

Helmitol.—Hexamethylenetetramine anhydromethylenecitrate, $C_6H_{12}N_4,C_7H_6O_7$.

Hemisine.—See Adrenaline.

Hermophenyl.—Mercury sodium phenoldisulphonate, $(SO_3Na)_2C_6H_2O(Hg)$.

Hetoform.—Basic bismuth cinnamate, $(C_6H_5.CH : CH.CO_2)_3Bi,Bi_2O_3$.

Hetralin.—Addition product of resorcinol and hexamethylenetetramine, $C_6H_{12}N_4,C_6H_4(OH)_2$.

Hexal.—Hexamethylenetetramine salicylsulphonic acid, $C_6H_{12}N_4,(HO)C_6H_3(COOH)SO_3H$.

Hexamine.—Hexamethylenetetramine, $C_6H_{12}N_4$.

Hexanatrin.—Combination of hexamethylenetetramine and acid sodium sulphate.

Hexanitrin.—Mannitol hexanitrate, $CH_2O(NO_2)(CHO.NO_2)_4.CH_2ONO_2$.

Homorenon.—Ethylaminoacetopyrocatechol hydrochloride, $(HO)_2.C_6H_3.CO.CH_2.NH(C_2H_5),HCl$.

Hopogan.—See Magnesium perhydrol.

Hydraldite A.—See Hydrosulphite N.F.

Hydraldite C.—See Hydrosulphite N.F. extra.

Hydramin.—A combination of *p*-phenylenediamine and quinol.

Hydrargol.—Mercury succinimide, $[C_2H_4(CO)_2N]_2Hg$.

Hydrargotin.—Mercury tannate (50 per cent. Hg).

Hydrarsan.—Solution of a mixture of arsenious, mercuric and potassium iodides and antipyrine.

Hydriodol.—See Cypridol.

Hydropyrin.—See Apyron.

Hydrosulphite N.F.—Condensation product of formaldehyde and sodium hydrosulphite; a mixture of formaldehyde sodium bisulphite, $NaHSO_3,CH_2O$, and formaldehyde-sodium sulphoxylate, $NaHSO_2,CH_2O$.

Hydrosulphite N.F. extra, or conc., or extra conc.—Formaldehyde-sodium-sulphoxylate.

Hyperol.—Compound of hydrogen peroxide and urea, with a trace of citric acid to render it stable.

Hypnogen.—See Veronal.

Iachiol.—Silver fluoride.

Ichthargan.—Silver ichthyolsulphonate (30 per cent. Ag).

Ichthermol.—Mercury compound of ichthyol.

Ichthosulphol.—Ichthyol.

Ichthynal.—See Ichthosulphol.

Ichthyolidin.—Piperazine ichthyolate.

Ichthyol-salicyl.—Mixture of ichthyol and sodium salicylate.

Imogen.—Sodium diaminonaphtholsulphonate, $(NH_2)_2C_{10}H_4(OH)SO_3Na$.

Indoform.—Salicylic methylene acetate; from formaldehyde and acetyl salicylic acid.

Insipin.—Diglycollic ester of quinine sulphate,
$(CH_2COO.C_{20}H_{23}N_2O)_2O,H_2SO_4,3H_2O$.

Iodanytol.—Ten per cent. solution of iodine in anytin (a 33 per cent. solution of ichthyol).

Iodeigon.—Albumin preparation containing iodine.

Iodin.—Iodised arachis oil.

Iodinol.—Iodised sesamé oil.

Iodival.—Monoiodoisovalerylurea, $(CH_3)_2CH.CHI.CO.NH.CO.NH_2$.

Iodocol.—Prepared from guaiacol and iodine in sodium iodine.

Iodofan.—Monoiododihydroxybenzene formaldehyde.

Iodoglidine.—Preparation of wheat gluten containing 10 per cent. I.

Iodoglobin.—Di-iodotyrosine, $HO.C_6H_2I_2.CH_2.CH(COOH)NH_2$.

Iodogluten.—Preparation of vegetable albumin containing 8 per cent. I.

Iodolen.—Combination of iodol (tetra-iodopyrrole) and albumin; contains 30 per cent. iodol.

Iodolin.—Quinoline chloromethyliodochloride, $(C_9H_7N(CH_3Cl)ClI$.

Iodolinum.—See Iodolin.

Iodolysin.—Preparation similar to tiodine; contains 43 per cent. thiosinamine and 47 per cent. I.

Iodomenim.—Iodised casein containing bismuth (10 per cent. I).

Iodophenin.—Tri-iodophenacetin.

Iodoprotein.—Albumin preparation containing 10 per cent. I.

Iodozol.—Di-iodo-*p*-phenolsulphonic acid, $C_6H_2I_2(OH)SO_3H,3H_2O$.

Iodylin.—Bismuth iodosalicylate.

Iodyloform.—Iodised gelatin (10 per cent. I).

Iohydrin.—Di-iodoisopropyl alcohol $CH_2I.CH(OH).CH_2I$.

Iothion.—See Iohydrin.

Isarol.—See Ichthosulphol.

Isoform.—*p*-Iodoxyanisol, $CH_3O.C_6H_4IO_2$.

Isopral.—Trichloroisopropyl alcohol, $CCl_3.CH(OH).CH_3$.

Istizin.—1.8.—Dihydroxy anthraquinone.

Jecovol.—Emulsion of cod-liver oil with glycerophosphates of sodium, calcium and iron.

Joha.—Solution of 40 per cent. of salvarsan in iodinol.

Kachin.—A pyrocatechol photographic developer.

Kakodyljacol.—See Cacodyliagol.

Kalmopyrin.—Calcium acetylsalicylate, $(C_2H_3O.OC_6H_4.COO)_2Ca$.

Kalzose.—Casein preparation containing calcium.

Kephaldol.—Preparation made from phenetidine and salicylic and citric acids.

Kinetine.—Combination of quinine and bectine.

Kineurine.—Quinine glycerophosphate, $C_3H_7O_3H_2PO_3(C_{20}H_{24}N_2O_2)_2,4H_2O$.

Koronium Bromide.—Strontium bromide, $SrBr_2,6H_2O$.

Krelos.—Mixture of tar distillates and rosin soap solution.

Kresegol.—See Cresegol.

Kresosteril.—See Cresosteril.

Kryogenin.—See Cryogenin.

Kynurin.—*p*-Hydroxyquinoline, $C_9H_6(OH)N$.

Lactagol.—Powdered extract of cotton-seed.

Lactanin.—Bismuth bilacto-monotannate.

Lacteol.—Preparation of lactic acid baccilli.

Lactigen.—See Lacteol.

Lactilloids.—See Lacteol.

Lactobaccilline.—See Lacteol.

Lactone.—See Lacteol.

Laxans.—See Purgen.

Laxatol.—See Purgen.

Laxen.—See Purgen.

Laxoin.—See Purgen.

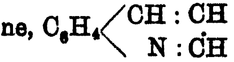
Lecithol.—Lecithin from egg yolk.

Lecutyl.—Combination of lecithin and copper cinnamate (1·5 per cent. Cu).

Lenicet.—Basic aluminium acetate, $Al_2(OH)_2(C_2H_3O_2)_4,H_2O$.

Lentin.—*m*-Phenylenediamine hydrochloride $C_6H_4(NH_2)_2,2HCl$.

Leptynol.—Colloidal palladous hydroxide suspended in sesamé oil.

Leucoline.—Quinoline, 
$C_6H_4\left\langle\begin{array}{l}CH:CH\\N:CH\end{array}\right.$

Levurargyre.—Mercury compound of nucleoprotein.

Levuretin.—Dried yeast cells.

Levurinose.—See Levuretin.

Lipobromol.—Brominated poppyseed oil (33 per cent. Br).

Lipoiodin.—Di-iodobrassidic acid ethyl ester, $C_{19}H_{30}CI : CI.CO_2C_2H_5$.

Lithium-Diuretin.—Addition product of theobrominelithium and lithium salicylate, $C_7H_7N_4O_2Li,HO.C_6H_4.COOLi$.

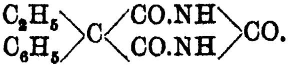
Lithyol.—See Ichthosulphol.

Lodal.—An oxidation product of laudanosine.

Lofotol.—Cod-liver oil charged with carbon dioxide.

Lucidol.—Benzoyl peroxide, $(C_6H_5.COO)_2O_2$.

Luesan.—Glidine preparation containing mercury.

Luminal.—Phenyl ethyl malonylurea, 
$\left.\begin{matrix}C_2H_5\\C_6H_5\end{matrix}\right\rangle C\left\langle\begin{matrix}CO.NH\\CO.NH\end{matrix}\right\rangle CO.$

Lycin.—See Acidol.

Lygosin.—Dihydroxydibenzalacetone $(HO.C_6H_4.CH:CH)_2CO$.

Lysochlor.—Chloro-*m*-cresol, $C_6H_3Cl(CH_3)OH$.

Lysoform.—A liquid formaldehyde potassium soap.

Magnesium-perhydrol.—Mixture containing magnesia and 15 or 25 per cent. MgO_2.

Malonal.—See Veronal.

Malourea.—See Veronal.

Maltyl.—Dry malt extract; contains about 90 per cent. of soluble carbohydrates.

Maretin.—*m*-Tolylsemicarbazide, $CH_3.C_6H_4.NH.NH.CO.NH_2$.

Maricol.—Magnesium ricinoleate, $(HO.C_{17}H_{32}CO_2)_2Mg$.

Medinal.—Sodium compound of veronal.

Melrubin.—Sodium 1-phenyl-2·3-dimethylpyrazolone-4-aminomethane sulphonate, $C_{11}H_{11}NO_2.NH.CH_2.SO_3Na$.

Melubrin.—Mercury salicylsulphonate.

Menthophenol.—Mixture of phenol 1, menthol 3 parts.

Mercuriocoleolo.—Double oleate of cholesterol and mercury.

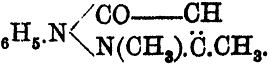
Mergal.—A combination of mercury cholate and albumin tannate.

Merjodin.—Mercury di-iodophenol-*p*-sulphonate, $(HO.C_6H_2I_2.SO_3)_2Hg$.

Merlusan.—Mercury compound of tyrosine (53 per cent. Hg), $C_9H_9O_3NHg$.

Merochinol.—Mercury hydroxyquinolinesulphonate, $(HO.C_9H_5N.SO_3)_2Hg$.

Mesotan.—Salicylic methoxymethyl ester, $HO.C_6H_4.COO.CH_2.OCH_3$.

Methozin.—Antipyrine (phenyldimethylpyrazolone), 
$C_6H_5.N\left\langle\begin{matrix}CO\text{——}CH\\N(CH_3).C.CH_3\end{matrix}\right.$

Methylrhodin.—Methyl acetylsalicylate, $C_2H_3O.O.C_6H_4.COOCH_3$.

Metoquinone.—Addition product of metol and quinol.

Metramine.—See Hexamine.

Migralgine.—Mixture of antipyrine 88, caffeine 9, and salicylic acid 3 parts, fused together.

Momordicine.—Elaterin, $C_{20}H_{28}O_5$.

Monotal.—Guaiacol methyl glycollate, $CH_2(OCH_3).COOC_6H_4.OCH_3$.

Morphosan.—Morphine methyl bromide, $C_{17}H_{19}O_3N.CH_3Br,H_2O$.

Mucogeène.—An anthraquinone derivative, $C_{18}H_{15}N_2O_2Cl$.

Mydriasine.—Atropine methyl bromide, $C_{17}H_{23}NO_3,CH_3Br$.

Naphtholsalol.—β-Naphthyl salicylate, $HO.C_6H_4.COO.C_{10}H_7$.

Narcyl.—Ethylnarceine hydrochloride, $C_{23}H_{26}(C_2H_5)NO_8,HCl$.

Nargol.—Silver nucleinate (10 per cent. Ag).

Nealpon.—See Pantopon.

Neoform.—Bismuth compound of tri-iodophenol, $C_6H_2I_3OBi(OH)_2,Bi_2O_3$.

Neoleptol.—Triformyltrimethylenetriamine.

Neopine.—Hydroxycodeine, $C_{18}H_{20}NO_3(OH)$.

Neopyrin.—Valerylaminoantipyrine.

Neosalvarsan.—Sodium 3·3′-diamino-4·4′-dihydroxyarsenobenzene-formaldehydsulphoxylate, $NH_2.C_6H_3(OH)As.As.C_6H_3(OH)NH(CH_2O)SONa$.

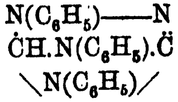
Neosiode.—Iodocatechin, $(C_{15}H_{14}O_6,3H_2O)_3I$.

Nerolin.—β-Naphthyl methyl ether, $C_{10}H_7O.CH_3$.

Neuraltein.—Sodium *p*-ethoxyphenylaminomethane sulphonate, $C_2H_5O.C_6H_4.NH.CH_2.SO_3Na$.

Neuronal.—Diethylbromo-acetamide, $(C_2H_5)_2CBr.CO.NH_2$.

Nitron.—1·4-Diphenyl-3·5-endanilodihydrotriazole,



$$\begin{array}{l} \quad N(C_6H_5)\text{——}N \\ CH.N(C_6H_5).C \\ \quad \diagdown N(C_6H_5)\diagup \end{array}$$

Nizin.—Zinc sulphanilate, $(NH_2.C_6H_4.SO_3)_2Zn$.

Novargan.—Protein preparation containing 10 per cent. Ag.

Novaspirin.—Methylenecitrylsalicylic acid.

Novatophan.—Ethyl 6-methyl-2-phenylquinoline-4-carboxylate, $C_6H_5.C_9H_4N(CH_3)COOC_2H_5$.

Noviform.—Bismuth compound of tetrabromopyrocatechol.

Novocaine.—*p*-Aminobenzoyldiethylaminoethanol hydrochloride, $NH_2.C_6H_4CO.O.CH_2.CH_2.N(C_2H_5)_2,HCl$.

Novocol.—Sodium monoguaiacol phosphate.

Nucleogen.—Preparation containing iron nucleinate and arsenic.

Oleanodyne.—Preparation containing oleic acid, aconitine, atropine, morphine, and veratrine.

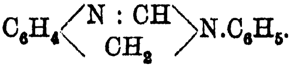
Oleoguaiacol.—Guaiacol oleate, $CH_3O.C_6H_4.O.CO.C_{17}H_{33}$.

Omnopon.—See Pantopon.

Omorol.—See Novargan.

Optochin.—Ethyl hydrocupreine.

Orexin.—Phenyldihydroquinazoline,



$$C_6H_4\left\langle\begin{array}{c}N:CH\\CH_2\end{array}\right\rangle N.C_6H_5.$$

Orsudan.—Sodium methyl acetyl-*p*-aminophenylarsinate, $C_2H_3O.NH.C_6H_3(CH_3).AsO(OH)(ONa)$.

Ortizon.—See Hyperol.

Ovoferrin.—Vitellin preparation containing iron.

Oxylith.—Sodium peroxide.

Oxyntin.—A combination of albumin and hydrochloric acid.

Palmiacol.—See Cetiacol.

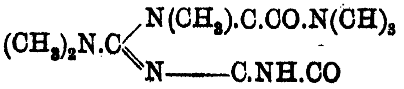
Pantopon.—Mixture of the soluble hydrochlorides of opium alkaloids.

Parabismut.—Bismuth paranucleinate.

Paracodine.—Dihydrocodeine, $C_{17}H_{19}(OCH_3)(OH)NO$.

Paralysol.—Solid cresol preparation, $C_6H_4(CH_3)OK,3C_6H_4(CH_3)OH$.

Paraxin.—Dimethylamino-1·7-dimethylxanthine,



$$(CH_3)_2N.C\left\langle\begin{array}{l}N(CH_3).C.CO.N(CH)_3\\N\text{——}C.NH.CO\end{array}\right.$$

Pegnine.—Preparation of lactose and rennet.

Pellidol.—Diacetylaminoazotoluene.

Pepsalia.—Preparation of pepsin and sodium chloride.

Perborax.—Sodium perborate, $NaBO_3,4H_2O$.

Perborol.—See Perborax.

Perhydrit.—See Hyperol.

Perhydrol.—Solution of hydrogen peroxide, 100 vol., or 30 per cent. by weight.

Permidan.—Dimethylaminopyrazolone, $C_5H_9N_3O$.

Phenalgin.—Phosphoammonioacetanilide.

Phenamine.—Amino-acet-*p*-phenetidine hydrochloride, $C_2H_5O.C_6H_4.NH.CO.CH_2.NH_2,HCl$.

Phenolax.—See Purgen.

Phenosal.—Monophenetidide of salicylacetic acid, $COOH.C_6H_4.O.CH_2.CO.NH.C_6H_4.OC_2H_5$.

Phenoval.—Bromovaleryl phenetidine, $(CH_3)_2CH.CHBr.CO.NH.C_6H_4OC_2H_5$.

Phenyform.—Condensation product of phenol and formaldehyde.

Phenylon.—See Methozin.

Phobrol.—Fifty per cent. solution of chloro-*m*-cresol in potash castor-oil soap.

Photoxylin.—See Celloidin.

Picrastol.—Dimethyloldiformylmethenyltetramethylenepentamine, $C_9H_{17}N_5O_4$.

Picratol.—Silver picrate.

Pinakol.—A pyrogallol photographic developer in which part of the alkali usually employed is replaced by sodium aminoacetate.

Piral.—Pyrogallol, $C_6H_3(OH)_3$.

Plecavol.—Consists chiefly of *p*-aminobenzoyleugenol, $NH_2.C_6H_4CO.O.C_6H_3(C_3H_5)OCH_3$, tricresol (a mixture of the three cresols), and formaldehyde.

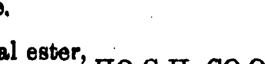
Pleistopon.—Similar to pantopon, except that it contains no narcotine.

Pneumin.—Condensation product of creosote and formaldehyde.

Pneumosan.—Amyl thiotrimethylamine.

Propæsin.—Propylester of *p*-aminobenzoic acid, $NH_2.C_6H_4.COOC_3H_7$.

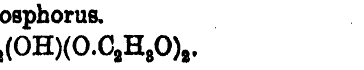
Proponal.—Dipropylbarbituric acid,



$$(C_3H_7)_2C{<}\begin{matrix}CO.NH\\CO.NH\end{matrix}{>}CO.$$

Proposote.—Creosote phenylpropionate.

Protosal.—Salicylic acid glycerol formal ester,



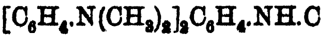
$$\begin{matrix} & O.CH_2O \\ HO.C_6H_4.CO.O.CH_2. & \dot{C}H-\dot{C}H_2.\end{matrix}$$

Protylin.—Albumin preparation containing phosphorus.

Purgatin.—Anthrapurpurin diacetate, $C_{14}H_5O_2(OH)(O.C_2H_3O)_2$.

Purgatol.—See Purgatin.

Purgen.—Phenolphthaleïn,



$$C_6H_4{<}\begin{matrix}C(C_6H_4.OH)_2\\OC\text{———————}\end{matrix}{>}O.$$

Pyoktanin.—Methyl violet, $[C_6H_4.N(CH_3)_2]_2C_6H_4.NH.CH_3)C.Cl$.

Pyracetosalyl.—Addition product of antipyrine and acetylsalicylic acid, $C_{11}H_{12}N_2O + C_2H_3O.OC_6H_4COOH$.

Pyrobromone.—Bromodimethylaminoantipyrine.

Pyronal.—Antipyrine salicylacetate, $C_{11}H_{12}N_2O,HO.C_6H_4CO.CH_2.COOH$.

Pyrophan.—Combination of pyrogallol and dimethylamine.

Quietol.—Dimethylaminodimethylisovalerylpropyl ester hydrobromide.

Quinaphenin.—Phenetidine quinine carbonic ester, $CO(NH.C_6H_4.OC_2H_5)(C_{20}H_{23}N_2O_2)$.

Quinaphthol.—Quinine 3-naphtholsulphonate, $C_{20}H_{24}N_2O_2,(HO.C_{10}H_6.SO_3H)_2$.

Quineonal.—Compound of quinine and veronal.

Quinine Lygosinate.—Dihydoxydibenzalacetone-quinine, $CO(CH:CH.C_6H_4.C_{20}H_{24}N_2O_2)_2$.

Quinoform.—Quinine formate, $C_{20}H_{24}N_2O_2,HCOOH$; also a condensation product of cinchotannic acid and formaldehyde.

Quinopyrin.—A combination of antipyrin 2, and quinine hydrochloride 3 parts.

Quinosol.—Mixture of hydroxyquinoline and potassium sulphates, $C_9H_6NOSO_3K,4H_2O$.

Quinotropin.—Hexamethylenetetramine quinate.

Regulin.—Broken-up agar-agar with 25 per cent. cascara extract.

Resaldol.—Resorcylbenzoic acid ethyl ester, $(HO)_2C_6H_3.CO.C_6H_4.CO_2C_2H_5$.

Rexotan.—Methylene tannin-urea.

Rheumatin.—Saloquinine salicylate, $HO.C_6H_4.COO.C_{20}H_{23}N_2O,C_6H_4(OH)COOH$.

Rhodaform.—Addition product of hexamethylenetetramine and methyl thiocyanate, $C_6H_{12}N_4,CH_3CNS$.

Riopan.—Soluble brown powder containing 50 per cent. of ipecacuanha alkaloids in the form of their hydrochlorides.

Rongalite C.—See Hydrosulphite N.F. extra.

Sabromine.—Calcium dibromobehenate, $(C_{22}H_{41}O_2Br_2)_2Ca$ (29 per cent. Br).

Sajodin.—Calcium monoiodobehenate, $(C_{22}H_{42}O_2I)_2Ca$.

Salacetin.—See Acetosal.

Saletin.—See Acetosal.

Salibromin.—Dibromosalicylic methyl ester, $C_6H_2Br_2(OH)COOCH_3$.

Salicreol.—Creosote salicylate.

Salimenthol.—Menthyl salicylate, $HO.C_6H_4.COOC_{10}H_{19}$.

Salit.—Bornyl salicylate, $HO.C_6H_4.COO.C_{10}H_{17}$.

Salocreol.—See Salicreol.

Saloquinine.—Solicylquinine, $HO.C_6H_4.COO.C_{20}H_{23}N_2O$.

Salufer.—Sodium silicofluoride.

Salvarsan.—Dihydroxydiaminoarsenobenzene dihydrochloride, $HO(NH_2.HCl)C_6H_3.As.As.C_6H_3(NH_2.HCl)OH,2H_2O$.

Salvarsan, New.—See Neosalvarsan.

Santheose.—Theobromine, $C_5H_2(CH_3)_2N_4O_2$.

Santyl.—Santalyl salicylate, $HO.C_6H_4.COO.C_{15}H_{23}$.

Sauerin.—Preparation of lactic acid bacilli.

Savore.—Preparation of milk, cereal proteins and albumoses, and carbohydrates.

Scopomorphine.—Sterilised solution containing, per c.c., 0·0006 grm. of euscopol and 0·015 grm. of morphine hydrochloride.

Sedeff.—Effervescent preparation containing opium, bismuth, and digestive ferments.

Sennatin.—Preparation containing the active principles of senna leaves.

Sennax.—Preparation containing the water-soluble glucosides of senna leaves.

Sidonal, New.—Mixture of quinic acid, $C_6H_7(OH)_4COOH$, and its anhydride.

Soamin.—Sodium arsanilate, $NH_2.C_6H_4.AsO(OH)(ONa),5H_2O$.

Solurol.—Thyminic acid (nucleotinphosphoric acid), $C_{30}H_{46}N_4O_{16}(P_2O_5)_2$.

Somnoform.—Mixture of ethyl chloride 60, methyl chloride 35, and ethyl bromide 5 per cent.

Sophol.—Silver formonucleinate (20 per cent. Ag).

Spirarsyl.—Sodium salt of arsenophenylglycine $(CO_2Na.CH_2.NH).C_6H_4)_2As_2$.

Spirosal.—Monoglycol ester of salicylic acid, $HO.C_6H_4.COO.CH_2.CH_2OH$.

Stearosan.—Compound of santalol and stearic acid.

Stovaine.—Hydrochloride of dimethylamino tertiary amyl benzoate,

$$C_6H_5COO.C(CH_3)(C_2H_5)CH_2N(CH_3)_2,HCl.$$

Styptol.—Cotarnine phthalate, $(C_{12}H_{13}NO_3)_2,C_6H_4(COOH)_2$.

Subcutin.—An æsthesin phenolsulphonate, $C_6H_4(COOC_2H_5)NH_2,HO.C_6H_4.SO_3H$.

Sublamine.—Mercuric ethylenediamine sulphate (43 per cent. Hg).

Sucramine.—Ammonium salt of saccharin.

Sulfoid.—Preparation of colloidal sulphur (80 per cent. S) with an albuminous substance as protective colloid.

Sulphofarin.—Triphenyl stibine sulphide, $(C_6H_5)_3SbS$.

Suprarenaline.—See Epinephrine.

Suprarenine.—See Adrenaline.

Synthalin.—Piperonylquinoline carboxylic acid methyl ester,

$$CH_2O_2.C_6H_3.C_9H_5N.COOCH_3.$$

Surgol.—Colloidal silver.

Tachiol.—Silver fluoride.

Tanargan.—A combination of tannin and silver albuminate.

Tanninphenolmethane.—Condensation product of tannin, formaldehyde, and phenol.

Tanninthymolmethane.—Condensation product of tannin, formaldehyde, and thymol.

Tannismuth.—Bismuth bitannate.

Tannisol.—Methyleneditannin, $(C_{14}H_9O_9)_2CH_2$.

Tannobromine.—A combination of dibromotannin and formaldehyde.

Tannocreosoform.—Condensation product of tannin, formaldehyde, and creosote.

Tannoguaiaform.—Condensation product of tannin, formaldehyde, and guaiacol.

Tannothymal.—See Tanninthymolmethane.

Tannyl.—Oxychlorocasein tannate.

Tenosin.—Solution containing, per c.c., 0·0005 grm. of β-iminazolylethylamine and 0·02 grm. of *p*-hydroxyphenylethylamine.

Testijodyl.—Combination of coagulated blood albumin and iodine.

Theobromose.—Lithium compound of theobromine, $C_7H_7N_4O_2Li$.

Theocin.—Sodium compound of theobromine, $C_7H_7N_4O_2Na$.

Theolactin.—Addition product of theobromine-sodium and sodium lactate.

Theosalin.—Addition product of theobromine-sodium and sodium sulphosalicylate.

Thephorin.—Addition product of theobromine-sodium and sodium formate,

$$C_7H_7N_4O_2Na,HCO_2Na,H_2O.$$

Thermiol.—Sodium phenylpropiolate, $C_6H_5.C : C.CO_2Na$.

Thialion.—Preparation containing sodium sulphate (26 per cent.), citrates of sodium (56), potassium (3) and lithium (3), sodium chloride (3) and water (9 per cent.).

Thigenol.—Sodium salt of ichthoform.

Thiophysem.—See Tiodine.

Tholaform.—Mixture of equal parts of menthol and trioxymethylene.

Throphleol.—Fifty per cent. solution of erythrophleine hydrochloride (from casca bark) in eugenol.

Thymatol.—Thymol carbonate, $CO(O.C_{10}H_{13})_2$.

Thymegol.—Mercury potassium salt of thymol-*p*-sulphonate.

Thymolin.—Mixture of naphthalene 18, camphor 1, and thymol 1 part.

Thymoloform.—Condensation product of formaldehyde and thymol.

Thyresol.—Santalol methyl ether, $C_{15}H_{23}O.CH_3$.

Tiodine.—Thiosinamine ethyl iodide, $C_3H_5.NH.CS.NH_2,C_2H_5I$.

Toxynon.—Sodium *m*-acetyl aminomercuribenzoate,

$$HgOH.C_6H_3(NH.C_2H_3O)CO_2Na.$$

Tricalcol.—Colloidal combination of tricalcium phosphate and albumin.

Triferrin.—See Ferrinol.

Trigemin.—Compound of butyl chloral hydrate and pyramidone (dimethylamino-antipyrine).

Trilactine.—Preparation of lactic acid bacilli.

Trinitrin.—Nitroglycerin.

Trixidin.—An emulsion of antimony tri-oxide (30 per cent. Sb_2O_3).

Tryen.—See Griserin novum.

Trygase.—Dried yeast-cells.

Trypan Red.—Prepared from tetrazotised benzidine sulphonic acid and 2 mols. of sodium naphthylaminedisulphonate,

$$(SO_3H)C_6H_3.N_2.C_{10}H_4(SO_3Na)_2NH_2C_6H_4.N_2.C_{10}H_4(SO_3Na)_2NH_2.$$

Tryparosan.—Chlorinated parafuchsin.

Tylcalsin.—See Kalmopyrin.

Tyllithin.—See Apyron.

Tylmarin.—Acetyl-*o*-coumaric acid, $C_2H_3O.OC_6H_4.CH : CH.CO_2H$.

Tylnatrin.—Sodium acetylsalicylate, $C_2H_3O.OC_6H_4.COONa$.

Tyramine.—*p*-Hydroxyphenylethylamine hydrochloride,

$$HO.C_6H_4.C_2H_4.NH_2,HCl.$$

Ulmarene.—See Mesotan.

Unal.—Alkali salt of *p*-aminophenolhydrochloride; *rodinal* is the concentrated solution.

Uraminoantipyrin.—Antipyrylurea, $NH_2.CO.NH.C_{11}H_{11}N_2O$.

Urapurgol.—See Helmitol.

Urea-Bromine.—Combination of urea and calcium bromide,

(36 per cent. Br),$4CO(NH_2)_2,CaBr_2$.

Uritone.—Hexamethylenetetramine.

Urocitral.—Addition product of theobromine-sodium and sodium citrate.

Urodonal.—Granular preparation of hexamethylenetetramine, sidonal (piperazine quinate), and lysidine (methylglyoxalidine).

Urogenin.—Addition product of theobromine and lithium hippurate, $C_7H_8N_4O_2,C_6H_5.CO.NH.CH_2.CO_2Li$.

Urol.—Urea quinate.

Urosin.—Lithium quinate, $C_6H_7(OH)_4COOLi$.

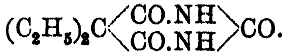
Urotropine, New.—See Helmitol.

Valamin.—Isovaleric ester of tertiary amyl alcohol.

Valearin.—Valeryltrimethylammonium chloride.

Valisan.—Bornylbromoisovalerate, $C_4H_8BrCO.OC_{10}H_{17}$.

Valyl.—Valeric diethylamide, $CH_3.CH_2.CH_2.CH_2.CO.N(C_2H_5)_2$.

Veronal.—Diethylbarbituric acid (diethylmalonylurea),  $(C_2H_5)_2C\left\langle\begin{matrix}CO.NH\\CO.NH\end{matrix}\right\rangle CO$.

Veropyrin.—Mixture of dionine (ethylmorphine hydrochloride) 0·01, veronal-sodium 0·2, and kalmopyrin, 0·5 part.

Vesalvine.—See Uritone.

Vesipyrine.—Phenyl acetylsalicylate, $CH_3.CO.OC_6H_4.COOC_6H_5$.

Vimopyrin.—*p*-Phenetidine tartrate.

Vioform.—Hydroxyquinoline chloroiodide, $C_9H_6N(OH)ICl$.

Vitafer.—Preparation of milk and glycerophosphates of calcium and sodium.

Xaxa.—See Acetosal.

Xaxaquin.—Quinine acetylsalicylate, $C_{20}H_{24}N_2O_2,CH_3CO.OC_6H_4.COOH$.

Yara-Yara.—See Nerolin.

Yatren.—See Griserin novum.

Zymin.—Special dry powdered yeast.

Occurrence of Methyl Alcohol in Corn Silage. E. B. Hart and A. R. Lamb. (*J. Amer. Chem. Soc.*, 1914, **36**, 2114-2118.)—Hart and Willaman (*J. Amer. Chem. Soc.*, 1912, **34**, 1619), concluded that methyl alcohol was present to the extent of about 0·05 per cent. on the silage mass, but Dox and Neidg (*Research. Bull.* No. 7, Iowa Exp. Sta.) failed to confirm this. The authors, using the methods of Trillat-Haigh and Sanglé-Ferrière-Cumiasse and the resorcinal phenol, gallic acid, and morphine tests, have shown that normal silage does undoubtedly contain small amounts of methyl alcohol. In all cases the tests were standardised by using various known mixtures of alcohols, and it is claimed that the number of different tests used precludes the possibility of positive reactions being given by substances other than methyl alcohol. About 300 to 400 grms. of the sample were comminuted in a food grinder, suspended in water, and distilled with steam under reduced pressure of from 30 to 40 mm. to prevent any possible chemical changes such as might occur at higher temperatures. About 1 litre was collected, thus obtaining rather less than half of the total volatile acids and alcohols originally present. The distillate, after neutralising with caustic soda, was redistilled under reduced pressure, and then repeatedly fractionated. Further experimental work showed that one source of the methyl alcohol was the action of micro-organisms present in the silage upon the glycine of the corn-proteins, both water cultures and experimental silage tests supporting this conclusion (*cf.* Ehrlich, *Wochenschr. Brau*, 1913, **30**, 561; and *Zeitsch. angew. Chem.*, 1914, **27**, 48). H. F. E. H.

Composition of "Dead" Rice. G. Silvestri. (*Annali Chim. Applic.*, 1914, 1, 212-213.)—The term "dead rice" (*grana morta del riso*) is given in Italy to immature and diseased grains. They are invariably present in unwashed rice, usually to the extent of about 5 per cent., though in certain unfavourable seasons (*e.g.*, 1912) the amount may average over 25 per cent., or in good harvests (*e.g.*, 1913) may fall to about 2 per cent. "Dead" rice is of a greenish-yellow colour, and has a wrinkled and irregular form. A sample had the following composition: Water, 15·01; ash, 1·74; nitrogenous substances, 7·18; starch, 66·4; and fat, cellulose, etc., 9·67 per cent.

C. A. M.

Test for Silicates in Soap. H. W. Leitch. (*J. Ind. and Eng. Chem.*, 1914, 6, 811-812.)—The test depends on the insolubility of aluminium silicate in a mixture of acetone and water. About 1 grm. of the soap is dissolved in 25 c.c. of water, hydrochloric acid is added, and the liberated fatty acids are separated by filtration. Ten c.c. of the clear filtrate, corresponding to about 0·3 grm. of soap, are treated with 5 c.c. of $\frac{N}{1}$ alcoholic potassium hydroxide solution, evaporated to 10 c.c., and this solution is poured into a test-tube containing 10 c.c. of acetone and 1 c.c. of a solution prepared by dissolving 10 grms. of sodium aluminate and 2 grms. of sodium chloride in 1 litre of water. If sodium silicate was present in the soap, a flocculent gelatinous precipitate is formed. Gelatin, starch, and dextrin, if present in the soap, interfere with the test; the former may be separated by treatment with alcohol or formaldehyde, and the two latter by digestion with diastase. A soap containing alkali carbonates, borax, sodium sulphate, cane-sugar, and dextrose, does not yield a precipitate with the acetone reagent; if a soap solution does not give a precipitate when added directly to the reagent, silicates are absent; but if a precipitate forms, the above procedure must be adopted to show if a silicate is actually present.

W. P. S.

Estimation of the Proteins in Wheat Flour. G. Silvestri. (*Annali Chim. Applic.*, 1914, 1, 214-215.)—The gluten from 33·33 grms. of the flour is triturated in a mortar with 70 per cent. alcohol containing 0·3 per cent. of potassium hydroxide, and the mixture (about 80 c.c. of liquid) vigorously shaken with glass beads to complete the disintegration of the gluten. After passage of a current of carbon dioxide the liquid is decanted, made up to 150 c.c., and filtered, 50 c.c. of the filtrate evaporated, and the residue (gliadin with a little potassium carbonate) dried at 105° C. and weighed. The potassium carbonate is determined in another portion of the filtrate and deducted to obtain the gliadin. The conglutin is determined by triturating the gluten with 0·3 per cent. potassium hydroxide solution, and adding sufficient alcoho to give an alcoholic strength of 70 per cent. The gliadin and conglutin are then precipitated together by means of sulphuric acid, and separated by washing the precipitate (by decantation) with slightly acidified 70 per cent. alcohol. The precipitate is next triturated with alkaline alcohol, and a current of carbon dioxide passed through the liquid, which is allowed to stand before filtration. The filtrate is treated with dilute sulphuric acid, and the precipitate of conglutin washed, dried, and weighed.

The glutenin is obtained by difference. Conglutin forms about 2 per cent. of the proteins of soft wheat, and about 20 per cent. of the proteins of hard wheat.

C. A. M.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Estimation of Carbohydrates and Lactic Acid in Blood and Other Physiological Liquids. L. Chelle. (*J. Pharm. et Chim.*, 1914, **106**, 319-322.)—The method is based on the colour reaction of the sugars with phenols in presence of sulphuric acid. For dextrose, 1 c.c. of the sugar solution and 5 c.c. of concentrated sulphuric acid are mixed, cooled, and treated with 0·2 c.c. of a 5 per cent. alcoholic solution of the phenol. With *a*-naphthol and thymol the reaction is sensitive to 5 mgrms. of dextrose per litre. For the detection of lactic acid in presence of dextrose codeïne may be used, since it gives no coloration with the sugar. The mixture of lactic acid solution and sulphuric acid, in the same proportions as above, is heated for two minutes in boiling water; the tube is immersed in cold water, and 0·1 c.c. of a 5 per cent. alcoholic solution of codeïne added. A straw-yellow coloration develops in seven or eight minutes. For the examination of blood, 1 c.c. of the sample and 1 c.c. of 5 per cent. sulphuric acid are added successively to 8 c.c. of a 0·6 per cent. solution of sodium metaphosphate, and the mixture is shaken and filtered. The quantitative estimation of lactic acid is performed by comparison with six standard tubes, each containing 1 c.c. of lactic acid solution ranging from 0·01 to 0·1 grm. per litre. A seventh tube contains 1 c.c. of the blood filtrate, and an eighth 0·5 c.c. of blood filtrate with 0·5 c.c. of water. The coloration is then produced by the addition of sulphuric acid and codeïne as described above. If the blood contains more than 1·5 grms. of dextrose per litre, the blood filtrate must be further diluted. For the estimation of dextrose by means of thymol an analogous series of standards is prepared, containing, however, only 0·5 c.c. of dextrose solution, with 0·5 c.c. of 0·1 per cent. sodium chloride solution. The seventh tube is charged with 0·5 c.c. of blood filtrate and 0·5 c.c. of water, whilst the eighth contains 0·25 c.c. of blood filtrate, 0·25 c.c. of sodium chloride, and 0·5 c.c. of water. The amount of lactic acid in the blood having been previously estimated, an equivalent amount of this acid should be added to the dextrose solutions in the standard tubes in order to produce shades of colour more directly comparable with that of the blood liquid. In the estimation of dextrose by means of *a*-naphthol this precaution is still more important.

J. F. B.

Comparison of Methods for the Determination of the Proteolytic Activity of Pancreas Preparations. J. H. Long and A. W. Barton. (*J. Amer. Chem. Soc.*, 1914, **36**, 2151-2166.)—A comparison of the proteolytic value of six pancreas preparations was made by four methods. It was hoped to find such relations as would permit the translation of activity as expressed on a given standard in terms of another. The activities of the six preparations were found by the four methods to be arranged in the same general order, but quantitatively their activity was very different, as will be seen by the table. Thus, the strongest ferment found by the metacasein reaction (No. 1) appears to be about twelve times the strength of the weakest and about ten times as strong by the fibrin digestion test (No. 4); by the other tests the relation is as two or three to one. It is not therefore possible to translate the proteolytic value of a tryptic ferment from the terms of one standard into the terms of another when dealing with the products furnished by chemical or pharmaceutical dealers, because these preparations are made by different processes of extraction, concentration, or activation, leaving mixtures of ferments in widely different proportions in the finished products, together with unknown amounts of inorganic salts. There is evidence to show that commercial trypsins or pancreatins contain at least two different enzymes of different degrees of thermostability, and reacting in different ways with proteins. The effects observed in any case are mixed effects, depending on the proportions in which the enzymes are present. The four methods employed were—(1) The metacasein method of Roberts (*Proc. Roy. Soc.*, 1886, **32**, 145), in which modified milk is employed as the source of protein. The unit of measurement is the number of mgrms. of milk converted to give the metacasein reaction in five minutes by 1 mgrm. of the ferment. (2) The Field-Gross method (*Arch. Exp. Path. und Pharm.*, 1906, **58**, 57), in which milk is replaced by a weak solution of sodium caseinate. The unit of measurement here is the number of mgrms. of casein digested in one hour by 1 mgrm. of ferment to the point at which no precipitate is given by the acetic acid reagent. (3) The Sörensen formaldehyde titration, a relatively strong solution of sodium caseinate being employed as the substrate. The unit is the relative amounts of alkali required to neutralise the amino-acids resulting from two hours' digestion of 1 grm. of casein. (4) The fibrin digestion method. Unit —mgrms. of fibrin digested to the soluble stage by 1 mgrm. of ferment in ten minutes. This method is probably the most unreliable, since after careful purification fibrin retains a little of the ferment which brings about fibrinolysis, resulting in gradual solution of the fibrin. Raw egg-albumen is unsuitable, since trypsin has little action on other than denatured proteins.

Ferment.	1. Metacasein Reaction.	2. Field-Gross Method.	3. Formaldehyde Titration.	4. Fibrin Digestion.
A	12,500	120	100	53·2
B	3,400	80	38	32·0
C	2,050	75	38	11·4
D	1,520	60	38	8·0
E	1,480	40	36	4·4
F	1,060	55	33	5·0

H. F. E. H.

Enzymes Present in Alfalfa (Lucerne). C. A. Jacobson and A. Holmes. (*J. Amer. Chem. Soc.*, 1914, **36**, 2170-2172.)—Aqueous extracts of the different parts of the plant were employed for the qualitative detection of the enzymes tabulated below.

The presence of the enzyme is denoted by +, its absence by −. If in small amount (*s*) is added, and if in large amount (*l*).

Enzyme.	Dried Plants.	Fresh Plants.	Fresh Roots.	Seeds.
Lipase	−	+(*s*)	−	−
Amylase	+(*s*)	+(*s*)	+(*s*)	+
Coagulase	−	+	+	+
Emulsin	+(*l*)	+(*l*)	+(*s*)	+
Invertase	+	+(*s*)	+	−
Peroxidase	+(*s*)	+	+(*l*)	+
Maltase	−			
Lactase	−			
Pectinase	+(*l*)	+(*l*)	+	
Protease (peptonising) ...	−	−	−	−
„ (peptolytic) ...	+	+	−	+

No digestion of egg-albumen could be detected by the proteases in any part of the plant, but this substance always retards the action of the enzyme on casein to an extent proportional to the time of the reaction, but not to the temperature.

H. F. E. H.

Manometric Method for the Estimation of Urea. W. Löb and A. Prorsk. (*Biochem. Zeitsch.*, 1914, **65**, 273-283.)—The liquid containing urea is decomposed with sodium hypobromite solution in an apparatus fitted with a manometer. The whole apparatus, with exception of the free arm of the manometer, is immersed in a thermostat, and the manometer read after the pressure has remained constant for ten to fifteen minutes. The apparatus is standardised by means of solutions of urea of known strength.

E. W.

Experiments with Castor Bean Urease. (Studies on Enzyme Action—XI.) K. G. Falk and K. Sugiura. (*J. Amer. Chem Soc.*, 1914, **36**, 2166-2170.)—Castor bean preparations were found to hydrolyse less urea than similar soy bean preparations under comparable conditions, indicating that the urease of castor beans is less active than that of soy beans, or that their urease content is less. In the experiments the castor or soy beans freed from oil were mixed with the urea and water or salt solution, and after the addition of a little toluene were allowed to incubate for various periods at 38° to 40° C. The ammonia formed was removed by blowing air through (ANALYST, 1913, **38**, 580) for two or three hours after sufficient 2N sodium hydroxide solution had been added to make the mixture alkaline to the extent of $\frac{N}{1}$, and titrated against standard acid. Dilute hydrochloric acid and sodium hydroxide solutions retard the action of the enzyme, while disodium phosphate exerts a small accelerating action. Magnesium and manganous sulphates cause very little change, unlike the action of these salts on castor bean lipase, which is much accelerated thereby. For shorter periods of time the amounts of urea decomposed by the enzyme are nearly proportional to the durations of action, but complicating reactions of a secondary character cause this relation to be departed from for longer periods.

H. F. E. H.

Volatile Substances of Urine. W. M. Dehn and F. A. Hartman. (*J. Amer. Chem. Soc.*, 1914, **36**, 2118-2136.)—The decomposition of complex non-volatile substances contained in fresh urine gives rise to malodorous volatile products not present in the original urine, but produced in it by hydrolysis, reduction, or oxidation. Some thirty-five volatile substances hitherto described as present in urine may be divided into four main groups: (1) acids, (2) phenols, (3) bases, and (4) neutral substances; which are again divisible into normal and abnormal constituents. The neutral volatile group, including "urinod" and three other new and closely related substances, contains the most important volatile substances of urine, among them being acetone, chloroform, iodoform, ethyl sulphide, methyl mercaptan, and the terpenes. Although some of these substances are contributary to the odour of urine, the newly-discovered "urinod" and urinod hydrosulphide is by far the most important agent (see following abstract). Fresh and fermented urines have lower vapour pressures than water, but at ordinary temperature the volatile acidity of urine is very minute. Very little volatile substance is obtainable by direct extraction of urine with ether, and the best results were got by distilling with dilute sulphuric acid and then extracting the distillates with ether and concentrating them. The principal volatile acids so obtained were benzoic (formed by hydrolysis of hippuric acid), hydrogen sulphide, the fatty acids up to heptylic acid, and possibly hexahydrabenzoic acid. The principal phenols were phenol and *p*-cresol; other higher phenols occur in notable quantities. Methylamine and indol occur in traces in fresh, and in larger quantities in fermented urines.

H. F. E. H.

"Urinod," the Cause of the Characteristic Odour of Urine. W. M. Dehn and F. A. Hartman. (*J. Amer. Chem. Soc.*, 1914, **36**, 2136-2146.)—In the preceding abstract reference is made to "urinod" as the principal agent causing the foul smell of stale urine. This substance can be prepared by treating urine with dilute sulphuric acid so as to make a 3 per cent. concentration of acid. After being allowed to stand for several days, the mixture, which is now dark-coloured and has an unpleasant odour, is distilled, and the distillates are extracted with ether, after which they are washed with sodium carbonate, sodium hydroxide, and hydrochloric acid, to remove acids, phenols, and bases. The residue is then distilled in steam, the distillate extracted with ether, and the ethereal solution shaken with mercury to remove sulphur; the solution, which is yellow or brown at this stage, is then concentrated and fractionated under reduced pressure. "Urinod" is a neutral malodorous substance with the empirical formula C_6H_8O. It occurs in urine in a combined form, and is liberated by fermentation and by treatment with dilute sulphuric acid. It appears to be present in all urines. It is a very toxic substance, and may bear some relation to uræmia.

H. F. E. H.

Detection of Peptides in Urine by the *p*-Cresol Tyrosinase Reaction. R. Chodat and R. H. Kummer. (*Biochem. Zeitsch.*, 1914, **65**, 392-400.)—The urine (3 c.c.), after being neutralised with sodium carbonate, is treated with 1 c.c. *p*-cresol (1 in 250) and 1 c.c. of a 0·5 per cent. tyrosinase solution, and shaken with toluene. With normal urine a red colour is produced after some minutes, which changes into green after some hours. This reaction is ascribed to the presence of peptides or polypeptides. Tyrosine or peptides containing tyrosine are absent in normal urine. Boiled urine gives the reaction more rapidly; this is due to the decomposition by heat of an unstable substance in urine which retards the reaction. E. W.

ORGANIC ANALYSIS.

Methods of Estimation of Carbohydrates. III.: The Cupric-Reducing Power of the Pentoses—Xylose and Arabinose. A. J. Daish. (*J. Agric. Sci.*, 1914, **6**, 255-262.)—Determinations have been made of the cupric-reducing powers of xylose and arabinose, carefully purified by recrystallisation and dried at 96° C. *in vacuo* over phosphorus pentoxide. The table below has been drawn up from the results obtained under the standard conditions defined by Brown, Morris, and Millar (*J. Chem. Soc.*, 1897, **71**, 72).

Cupric-Reducing Powers of Xylose and Arabinose.

Mgrms. Sugar.	Xylose, $[\alpha]^{20}_{D} = +18·78°$ ($c = 5·0755$).			Arabinose, $[\alpha]^{20}_{D} = +102·14°$ ($c = 6·8064$) $= +102·33°$ ($c = 4·669$).		
	Grm. CuO.	Calculated Divisor.	Divisor from Curve.	Grm. CuO.	Calculated Divisor.	Divisor from Curve.
10	0·0280	2·800	2·656	0·0270	2·700	2·669
20	0·0540	2·700	2·638	0·0540	2·700	2·654
30	0·0798	2·660	2·620	0·0804	2·680	2·640
40	0·1040	2·600	2·602	0·1064	2·660	2·625
50	0·1300	2·600	2·581	0·1320	2·640	2·610
60	0·1540	2·583	2·563	0·1570	2·617	2·595
70	0·1790	2·557	2·545	0·1820	2·600	2·581
80	0·2030	2·537	2·526	0·2060	2·575	2·566
90	0·2260	2·511	2·508	0·2300	2·556	2·551
100	0·2490	2·490	2·490	0·2540	2·540	2·536
110	0·2720	2·473	2·471	0·2780	2·527	2·521
120	0·2940	2·450	2·453	0·3020	2·517	2·507
130	0·3160	2·431	2·433	0·3248	2·499	2·492
140	0·3380	2·414	2·415	0·3476	2·483	2·477
150	0·3600	2·400	2·397	0·3700	2·467	2·461
160	0·3810	2·381	2·378	0·3920	2·450	2·447
170	0·4020	2·365	2·360	0·4140	2·435	2·432
180	0·4230	2·350	2·341	0·4360	2·422	2·417
190	0·4440	2·337	2·322	0·4570	2·405	2·403
200	0·4640	2·320	2·304	0·4780	2·390	2·381

To calculate the weight of sugar corresponding to any given weight of CuO, this latter should be divided by the divisor deduced from the curve, this being more accurate than that calculated from the individual results. For an unknown mixture of pentoses the average values for xylose and arabinose may be used without appreciable error; it will be noted that the cupric-reducing powers of these pentoses are very similar to that of dextrose. J. F. B.

Estimation of Aldoses (Part I.). Action of Neutral Sodium Hypoiodite. N. Bland and L. L. Lloyd. (*J. Soc. Chem. Ind.*, 1914, **33**, 948-949.)—For the estimation of formaldehyde, the alkaline hypoiodite method gives results agreeing well with those obtained by the hexamethylenetetramine method. It is not successful with paraformaldehyde, and gives low and variable results with acetaldehyde, owing possibly to the formation of iodoform. When applied to solutions of sugars, the results vary according to the quantity of alkali present. The author has therefore worked with a neutral solution of hypoiodite obtained by mixing 50 c.c. of $\frac{N}{10}$ iodine with 50 c.c. of $\frac{N}{10}$ sodium hydroxide solution. To this mixture were added 25 c.c. of 1 per cent. solutions of various sugars, the liquid being allowed to remain for about five minutes before acidification. The results obtained when calculated in molecular ratios showed—Dextrose, 96·4 to 99·7; lactose, 99·1 to 99·4; maltose, 99·9 to 101·8—per cent.; lævulose and cane-sugar, *nil*. The sugars after inversion by acid and neutralisation showed: Pure cane-sugar, 100·8 to 101·3; ordinary cane-sugar, 102·6 to 103·4; lactose, 99·1 to 99·4; maltose, 99·1 to 99·9—per cent. J. F. B.

Volumetric Estimation of Carbon in Aliphatic Substances in the Wet Way. E. C. Grey. (*J. Chem. Soc.*, 1914, **105**, 2204.)—The substance is oxidised by heating it for thirty minutes with about eight times its weight of a mixture of phosphoric acid of sp. gr. 1·75 and potassium dichromate. Most aliphatic compounds are oxidised to carbon dioxide, or to acetic acid and carbon dioxide, one molecule of acetic acid being produced from each carbon-connected methyl group. The carbon dioxide is measured, and the acetic acid distilled off in steam and titrated. The method has the advantage of giving some clue to the constitution of the compound. Some compounds, such as carbamide and succinic acid, require three hours' heating for complete oxidation. O. E. M.

"Lacmosol," the Active Constitent of Lacmoid. R. Hottinger. (*Biochem. Zeitsch.*, 1914, **65**, 177-189.)—Commercial lacmoid consists mainly of three different colouring matters, the colour changes of which are to some extent antagonistic. Lacmosol, which is present to the extent of about 20 per cent. in lacmoid, can be isolated from the latter, but is best prepared by heating 10 grms. resorcinol with 2 c.c. of a concentrated sodium nitrite solution for forty minutes at 105° C. The products of the reaction are dissolved in water and poured into 1 litre of a concentrated salt solution acidified with hydrochloric acid. The precipitate is collected and washed with brine, and may be purified by dissolving in ammonia and reprecipitating with hydrochloric acid. The precipitate is then dissolved in a small quantity of acetone or alcohol and added to about thirty times its volume of ether. The lacmosol remains dissolved in the ether, whilst lacmoid is precipitated. The indicator can be applied in aqueous, ethereal and alcoholic solution. Lacmosol is superior to lacmoid with respect to sensitiveness, purity of colour change, and constancy of composition. It is especially recommended for use in nitrogen estimations (Kjeldahl).

E. W.

Standard Tests for Lubricants. (Advance proof, *Amer. Soc. Testing Materials*, June-July, 1914.)—The following methods are recommended by the majority report of Committee D 2: Viscosity by the Saybolt viscometer; sp. gr. by the hydrometer, Westphal balance, or Geissler pyknometer; flash-point by the Cleveland cup or Pensky-Martens apparatus; detection of soap by the alcoholic metaphosphoric acid test; saponification value; free acids by titration of an alcoholic solution with potassium hydroxide; sulphur by combustion with oxygen under 30-40 atmospheres in the calorimetric bomb in presence of water, the sulphuric acid being determined as barium sulphate; water by distilling with xylene, or adding benzene, and measuring the water; precipitate formed on adding 10 c.c. of the oil to 90 c.c. of gasoline; microscopic detection of carbon, paraffin, and other solid impurities; carbon residue by distilling from a silica flask until all volatile matter is removed. A minority report recommends the retention of the Engler viscometer as an alternative.

O. E. M.

Examination of China Wood Oil. E. E. Ware and C. L. Schumann. (Advance proof, *Amer. Soc. Testing Materials*, June-July, 1914.)—*First Method:* The oil is diluted with five times its volume of petroleum ether and cooled to 0° C. Iodine added as ice-cold saturated solution in petroleum ether catalytically accelerates the action of light in converting the oil into a compound insoluble in petroleum ether. The precipitate is filtered off three times at intervals of three hours, when 93 per cent. of the oil should be precipitated. Any excess over 7 per cent. unprecipitated is to be regarded as adulterant. *Second Method:* The oil is saponified with absolute alcoholic potash, and the resulting soap, being very slightly soluble in cold absolute alcohol, separates, and is filtered off, washed with absolute alcohol which has been saturated at 0° C. with the soap, dried at 80° C., and weighed.

O. E. M.

Hydrogen Value of Some Essential Oils and Essential Oil Products. I. Oils of Sassafras, Anise, Fennel, Clove, and Pimento. A. R. Albright. (*J. Amer. Chem. Soc.*, 1914, **36**, 2188-2202.)—Results are given of an investigation regarding the hydrogenisation of essential oils in the presence of colloidal palladium; the hydrogen value of an oil is the number of c.c. of hydrogen at 0° C. and 760 mm. pressure absorbed by the oil during the period of most rapid absorption of the gas. The catalyst and alcohol are placed in a glass vessel filled with hydrogen, a weighed quantity of the oil is introduced, and, while the vessel is agitated continuously, hydrogen is admitted from a measuring burette; a decided and abrupt decrease in the rate of absorption of the gas denotes the end of the reaction. The following hydrogen numbers were obtained with various oils: Saffrol, 135·6; imitation sassafras oil, 111·9; genuine sassafras oil, 102·0; anethol, 150·5; imitation anise oil, 125·1; commercial anise oil, 125·8; commercial fennel oil, 101·3; eugenol, 134·4; imitation clove oil, 113·2; commercial clove oil, 114·0; commercial pimento oil, 97·8.

W. P. S.

Volatile Oil of *Calycanthus Floridus*. E. R. Miller, G. W. Taylor, and M. H. Eskew. (*J. Amer. Chem. Soc.*, 1914, **36**, 2182-2187.)—This member of the plant family *Calycanthaceæ* was obtained from Alabama; the twigs, free from leaves, were found to yield from 0·25 to 0·53 per cent. of their weight of volatile oil, the smaller, younger plants giving the best yield. Several specimens of the oil were prepared and examined; they were of a light yellow colour and had an aromatic odour suggestive of cineol. The sp. gr. at 25°/25° C. varied from 0·9209 to 0·9161; rotation in a 100 mm. tube, +2·85 to +6·6; refractive index at 26° C., 1·4675 to 1·4753; saponification value, 12·5 to 16·6; saponification value of acetylated oils, 65·7 to 75·1. The oils contained from 4·37 to 5·81 per cent. of esters, calculated as bornyl acetate, and from 14·46 to 18·44 per cent of free borneol. The cineol content varied from 35 to 70 per cent., the oil from the older plants containing the smaller quantity, and apparently a higher percentage of pinene. The oils also contained *d*-α-pinene, *l*-α-pinene, linalool (?), salicylic acid, and one or more esters in addition to bornyl acetate.

W. P. S.

Essential Oil of *Citrus Sinensis*. P. Fenaroli. (*Annali Chim. Applic.*, 1914, **1**, 408-412).—The essential oil distilled from the peel of the Chinese orange (*Citrus Sinensis*) of the Ligurian coast is principally used for the adulteration of oils of orange and lemon. Its orange odour is accompanied by another unpleasant odour which cannot be eliminated, and prevents its being used alone. Various samples gave the following analytical results: Sp. gr. at 15° C., 0·847 to 0·848; $\alpha[_D]_{20}$, 94·10 to 95·40; $[N]_{D\,17°C.} = 1·47388$ to 1·47408; solubility in 90 per cent. alcohol, 4 to 4·5 vols. When distilled under reduced pressure (80 mm.) it yielded the following fractions:

°C.	Grms.	$[\alpha]_D$ 14° C.
102-104	125	96·76
104-105	55	98·80
105-110	10·5	96·28
110-120	4	80·64
residue	5·5	23·40

The oil consisted in the main (97 to 98 per cent.) of *d*-limonene, with traces of a compound of lower b.-pt. (probably *d*-pinene), and esters and aldehydes boiling above 176° C. No citral could be detected.

C. A. M.

Oils of the *Coniferæ*. Leaf and Twig, and Bark Oils of White Fir. A. W. Schorger. (*J. Ind. and Eng. Chem.*, 1914, **6**, 809-810.)—The leaves and twigs of the white fir, *Abies concolor*, yield from 0·029 to 0·272 per cent. of oil, having sp. gr. at 15° C., 0·8720 to 0·8777; $[N]_{D\ 15^\circ C.}$, 1·4781 to 1·4796; $[\alpha]_{D\ 25^\circ C.}$, −20·11 to −27·94; acid value, 1·01 − 1·81; ester value, 12·52 to 27·34. The bark of the tree yields 0·1 per cent. of oil, having sp. gr. at 15° C., 0·8702 to 0·8767; $[N]_{D\ 15^\circ C.}$, 1·4809 to 1·4833; $[\alpha]_{D\ 20^\circ C.}$, −20·15 to −20·95; acid value, 0·87 to 1·22; ester value, 6·43 to 6·88. The oils have the following approximate composition:

	Leaf and Twig Oil.	Bark Oil.
Furfural	Trace	Trace
l-α-Pinene	12	9
l-Camphene	8	—
l-β-Pinene	42	60
l-Phellandrene	15	Dipentine 12 to 13
Esters as bornyl acetate ...	6·5	2·5
Free borneol	9·5	4·5
"Green oil"	3	5
Losses	4	7

The "green oil" was a fraction obtained after the removal of the esters; it boiled at 265° to 285° C., and exhibited a strong green fluorescence.

W. P. S.

Investigations on Oil of Black Sage. C. E. Burke and C. C. Scalione. (*J. Ind. and Eng. Chem.*, 1914, **6**, 804-806.)—The leaves and twigs of black sage, *Ramona Stachyoides*, a shrub growing in Southern California, yield 0·90 per cent. of their weight of oil. The authors have estimated approximately the number of constituents in the oil and their relative quantities, the method adopted consisting in fractionally distilling the oil, using a Hempel column, and plotting the following curves: (*a*) Temperature of distillation against the percentage weight which the fraction distilling at the temperature bears to the total; (*b*) specific gravity against percentage weight; (*c*) refractive index against percentage weight. At least five components were indicated by these means, and the various fractions were further treated with the object of isolating and identifying their chief constituents. The oil was found to consist of pinene, 6·0; cineol, 30·0; dipentene, terpinene, etc., 25·0; thujone, 8.0; camphor, 25·0; resinous substances, 5·0 per cent.

W. P. S.

Estimation of Phosphoric Acid in the Presence of Phosphoric Acid Esters. W. Heubner. (*Biochem. Zeitsch.*, 1914, **64**, 401-409.)—Phosphoric acid is quantitatively precipitated as ammonium phosphomolybdate in six hours at 37° C. from a solution containing 4 per cent. sulphuric acid (by volume) and 15 per cent. ammonium nitrate. Small concentrations of phytin do not interfere, but increasing concentrations of phytin hinder the precipitation. In the presence of a quantity of phytin equivalent to six times the amount of inorganic phosphorus, and at a concentration of 0·05 per cent., three-quarters of the inorganic phosphorus is precipitated. Glycerol phosphoric acid affects the precipitation to a lesser extent.

E. W.

Volumetric Method for the Estimation of Phytin. W. Heubner and H. Stadler. (*Biochem. Zeitsch.*, 1914, **64**, 422-438.)—Phytin can be estimated by titration of its solution containing 0·6 per cent. hydrochloric acid with a ferric chloride solution containing the same amount of hydrochloric acid and 0·05 to 0·2 per cent. of iron, using ammonium thiocyanate (0·03 per cent.) as indicator. One mgrm. iron corresponds to 1·19 mgrm. phytin-phosphorus. The titration can be carried out in the presence of inorganic phosphates and phosphoric acid esters which are not precipitated by ferric chloride in acid solution, provided that these substances be not present in too large an excess.

E. W.

Quantitative Determination of Body and Solvent in Varnish. A. L. Brown. (Advance proof, *Amer. Soc. Testing Materials*, June–July, 1914.)—The varnish is mixed with about ten times its volume of a volatile solvent such as ether. The solvent is evaporated on a ground-glass plate at the ordinary temperature in a stream of coal-gas. The original solvent in the varnish is carried off with the vapour of the added solvent, and the weight of film gives the quantity of "body," or non-volatile matter in the varnish taken. The drying qualities of the varnish may be tested on this film by observing the rate at which it takes up oxygen on exposure to air. Examples showing a comparison of two insulating varnishes are given.

O. E. M.

Waxes from Flax and Hemp. A. Bianchi and G. Malatesta. (*Annali Chim. Applic.*, 1914, 1, 297-302.)—Flax fibres of different origin yielded on extraction with carbon tetrachloride 2 to 2·5 per cent. of a wax-like fat with the following values: Sp. gr. at 15° C., 0·9980; m.-pt., 68° to 70° C.; acid value, 23 to 23·8; ester value, 132 to 139; and iodine value, 17 to 17·5. The unsaponifiable matter (20 to 23 per cent.) was a brown fragile substance melting at 73° to 74° C., and having a sp. gr. of 0·986. It consisted mainly of hydrocarbons, and did not contain phytosterols.

Hemp Wax.—Different samples of Italian origin yielded 0·5 to 1·2 per cent. of a brownish wax with the following characters: Sp. gr. at 15° C., 1·019; m.-pt., 66° to 67° C.; acid value, 46 to 47; ester value, about 145; and iodine value, 22·2 to 22·8. The unsaponifiable matter (11 per cent.) melted at 60° C., had a sp. gr. of 1·0022, and closely resembled the flax waxes.

C. A. M.

INORGANIC ANALYSIS.

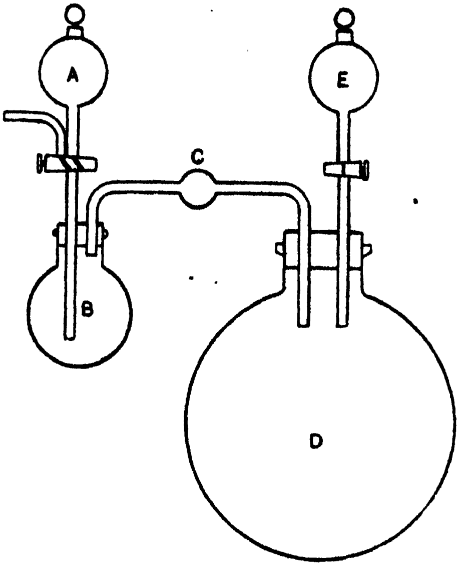
Determination of Carbon in Steel by the Direct Combustion Method. W. Brady. (*J. Ind. and Eng. Chem.*, 1914, **6**, 843-845.)—The paper describes and illustrates apparatus for the simultaneous analysis of seven samples by that modification of the direct combustion method in which the carbon dioxide is absorbed in a measured volume of barium hydroxide solution of known strength. The precipitated barium carbonate and unchanged hydroxide solution are rinsed into a flask, and the excess of hydroxide is titrated with hydrochloric acid, using phenolphthaleïn as indicator.

The only purifying train required before the furnace is a wash-bottle containing a solution of potassium hydroxide (D 1·27), and an 8-inch tower of soda lime. Between the furnace and the absorption apparatus is a bubble tube containing granulated zinc of 20 mesh to retain any sulphur trioxide. The nickel or platinum boats are lined with "RR alundum, 60 mesh, alkali free, specially prepared for carbon determinations." Platinised asbestos mixed with broken pieces of quartz tubing is used as catalyst just in front of the boat. The remainder of the forward part of the tube is filled with broken quartz, and the stopper at the back end is kept cool by a plug of asbestos contained in a cylindrical platinum basket which is attached to the stopper, so as to be removable with it for the insertion and removal of boats. Electric furnaces are illustrated, but the author uses gas furnaces also, the usual temperature being 960° to 1,000° C., though very low carbon steels or coarse drillings may require 1,100° C. Quartz tubes 24 inches by $\frac{3}{4}$ inches are used. Nickel boats must be thoroughly burned in oxygen until they give no blank before being used.

The absorption apparatus is constructed from a 500 c.c. conical flask and a 20-inch piece of $\frac{3}{8}$-inch glass tube on which nine 1-inch bulbs are blown as close as possible to each other, beginning near one end. The plain portion of the tube is passed through one of the two holes in the rubber stopper of the conical flask, and then bent parallel to the side of the flask. The second hole in the rubber stopper carries a small inlet tube connecting to the exit of the zinc bubble-tube. The absorption flasks are charged with 80 c.c. of 1·5 per cent. barium hydroxide solution, and inclined so that the bent stem of the bulb tube is vertical. The flask then functions as the large bulb of a Meyer tube, and the gases force the solution into the bulbs. At the end of a determination the flask is set vertically, the bulb tube rinsed down from above, and its stem also rinsed externally, the rinsings being caught in the flask, which is removed for titration and replaced by another charged with barium hydroxide for the next determination. A combustion occupies about thirty minutes, and requires no attention.

G. C. J.

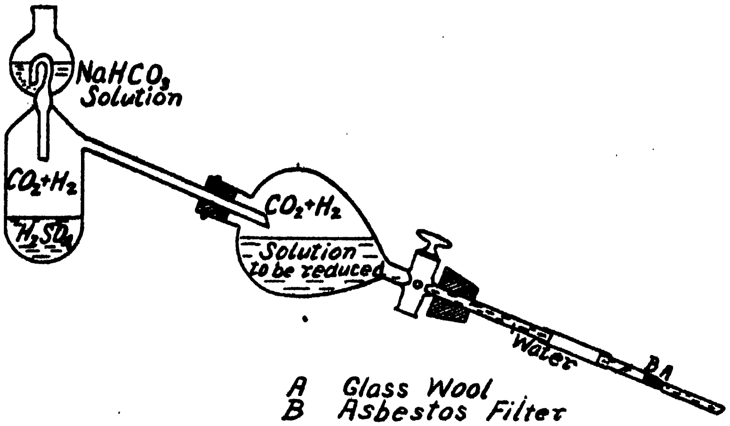
Analysis of a Sample of Monel Metal. (*Chem. News*, 1914, **110**, 196.)—To 0·5 grm. of the sample dissolved in the smallest possible quantity of nitric acid were added 5 c.c. of concentrated sulphuric acid. After evaporation, until fumes of sulphur trioxide were evolved, the liquid was diluted to 100 c.c., and 50 c.c. of strong sulphur dioxide solution and about 2 grms. of potassium iodide added. The precipitated cuprous iodide was washed with alcohol and ether, dried at 105° C., and weighed. The filtrate was boiled to remove sulphur dioxide, and about 1 grm. of ammonium persulphate, and dilute ammonium hydroxide sufficient to insure alkalinity, were added. The precipitated ferric and manganic hydroxides were filtered off, redissolved, reprecipitated, ignited, and weighed as $Fe_2O_3 + Mn_3O_4$, fused with potassium hydrogen sulphate, extracted with water and dilute sulphuric acid; the ferric sulphate was reduced with zinc and titrated with permanganate. The combined filtrates from the iron and manganese were made up to 1,000 c.c., and 250 c.c. were boiled with excess of a 6 per cent. solution of dimethylglyoxime in alcohol, and ammonia added. The precipitate was collected on a hardened filter, washed, dried in a platinum dish at 100° to 105° C., and weighed as $(C_4H_7N_2O_2)_2Ni$. O. E. M.



Determination of Soil Carbonates. H. B. Hutchinson and K. MacLennan. (*J. Agric. Sci.*, 1914, **6**, 323-327.)—In the estimation of carbonates in soil the error caused by the evolution of carbon dioxide by the decomposition of the organic matters may be prevented by avoiding the use of strong acids and high temperatures. The apparatus devised by the authors is shown in the illustration: Two round-bottomed, bolt-head flasks, *B* and *D*, of 100 and 1,000 c.c. capacity, respectively, and fitted with rubber stoppers. are connected by a stout bulb-tube, *C*, The flask *B* is fitted with a dropping funnel, *A*, with three-way stopcock, as shown, while *D* carries an ordinary dropping funnel, *E*. The sample is placed at the bottom of flask *B* and 50 c.c. of 2 per cent. hydrochloric acid in the funnel *A*. The funnel *E* is charged with 50 c.c. of $\frac{N}{10}$ sodium hydroxide solution and the stopper replaced. The apparatus is then exhausted by connecting it with a pump capable of giving an internal pressure of 60 to 100 mm. of mercury. The tap *A* is closed, and the sodium hydroxide admitted to the flask *D*, leaving 1 to 2 c.c. of the alkali in the funnel to absorb any carbon dioxide which may leak through the tap. Acid is then run into the flask *B*, allowing the main reaction to subside before agitating the apparatus. When the evolution of gas is finished (about twenty minutes), washed air is allowed to bubble in through the side-tube of *A*, with occasional agitation to facilitate absorption of the gas. After twenty to sixty minutes the flask *D* is disconnected and the residue of alkali in the funnel washed down into the flask. The carbon dioxide may be estimated by double titration with phenolphthaleïn and methyl orange, but since the bicarbonate point with the former is somewhat uncertain, the authors prefer to add an excess of barium chloride to the alkali just before titration, and titrate the excess of free hydroxide in presence of phenolphthaleïn; the precipitate of barium carbonate may subsequently be titrated in presence of methyl orange as a confirmation of the result. Estimations made in this apparatus with known quantities of calcium or sodium carbonate give perfectly satisfactory results.

J. F. B.

Volumetric Estimation of Titanium by Means of Ferric Chloride. T. R. Ball and G. McP. Smith. (*J. Amer. Chem. Soc.*, 1914, **36**, 1838-1843.)—The method proposed is an application of that described by Knecht and Hibbert for the estimation of iron. The titanium solution is first reduced by means of zinc in the apparatus shown; the valve device on the left prevents air from entering the funnel containing the zinc and the solution to be reduced. An excess of zinc is employed, and the reduction is allowed to proceed for two hours, after which the valve is removed from the neck of the funnel and the solution is drawn through the asbestos and glass-wool filter (*AB*) into a suction-flask previously filled with carbon dioxide. The funnel is washed with freshly boiled water in such a way that air is not permitted to enter the filter-tube, the end of which should reach to the bottom of the flask. Potassium thiocyanate solution is then added to the filtrate, and this is titrated as rapidly as possible with standardised ferric chloride solution. Ilmenite and other titaniferous iron ores should be finely powdered and then fused in platinum with a mixture consisting of 1 part, by weight, of potassium fluoride and 6 parts of potassium pyrosulphate. The fused mass, when cold, dissolves readily in hydrochloric acid, giving a clear solution which is ready for reduction. The modified valve shown answers better than the ordinary Contat-Gockel valve; it should be filled with carbon dioxide just before being attached to the funnel. The solution to be reduced must not contain such metals as tin, chromium, vanadium, tungsten, and molybdenum. W. P. S.



Oxidation of Sulphur Compounds of Coal, and of Nitrogen in the Bomb Calorimeter, and the Correction to be applied in determining the Heating Value of Coal. S. H. Regester. (*J. Ind. and Eng. Chem.*, 1914, **6**, 812-822.)—It is generally assumed that the sulphur of coal is oxidised almost quantitatively to sulphur trioxide in the bomb calorimeter. From the amount of sulphuric acid in the bomb washings it is usually considered that the sulphur content of the coal may be calculated, and as the sulphur is only oxidised to dioxide in boiler furnaces and the like, it is customary to make a deduction from the heat value as determined by the bomb calorimeter. Yet, from what is known of the equilibrium of sulphur dioxide, oxygen and sulphur trioxide, it does not seem probable that the sulphur of coal would burn completely to sulphur trioxide in the bomb calorimeter, unless some catalytic or oxidising agent were present.

The experimental work described in this paper was directed to determining whether the sulphur of coal is completely burned to trioxide in the bomb calorimeter, and, if so, what conditions are responsible for complete conversion from the dioxide. The chief conclusions are that oxidation to trioxide is, as a rule, almost complete (95 to 98 per cent.), and that this is due to the action of oxides of nitrogen, derived from the fuel or the commercial oxygen, which act as in the lead-chamber process. The pressure of the oxygen in the bomb favours the oxidation, but the influence of this factor is small. The ash exercises no measurable catalytic action.

The influence of oxides of nitrogen is illustrated by many experiments. When benzoic acid (or other nitrogen-free organic substance) is burned with pyrites in high-grade (99·5 per cent.) oxygen, only about half the sulphur is oxidised to trioxide. When "oxygen" containing 5 per cent. of nitrogen is used, or when the benzoic acid and pyrites are mixed with a sufficient amount of certain nitrogenous substances, the oxidation is nearly quantitative. None of the solid nitrogenous substances tried, however, was so effective as the nitrogenous matter of coal itself, and it is shown that it is the "fixed" nitrogen (that which remains in the coke) which is most effective. Consequently, the oxidation of the sulphur in coal to sulphur trioxide will usually be nearly quantitative, however the calorimetric combustion be carried out. With coals very low in nitrogen, however, there is a risk of the proportion of sulphur dioxide greatly exceeding the usual 2 to 5 per cent., unless care be taken to have 5 to 6 per cent. of nitrogen present in the oxygen. This proportion will always be present if the air in the bomb is not displaced by oxygen, as has been recommended by the Committee on Coal Analysis. The author, consequently, opposes this recommendation. In oxygen containing 6 per cent. of nitrogen, and under 20 atmospheres pressure, the sulphur in any organic substance, even if a nitrogen-free substance, may be oxidised almost quantitatively, but not more than 80 mgrms. sulphur should be present if a 350 c.c. bomb is used, or there will be too little nitric acid formed to bring about complete oxidation. G. C. J.

Method for the Estimation of Zinc in Treated Wood. M. H. Bedford and R. Pfanstiel. (*J. Ind. and Eng. Chem.*, 1914, **6**, 811.)—Several methods have been proposed for the destruction of the organic matter previous to the estimation of zinc in wood which has been treated with zinc chloride, but none of these methods yields satisfactory results in the case of hard woods; for instance, in dealing with red oak there is great difficulty in destroying all the organic matter. The authors therefore recommend the following process in which the wood is mixed with sodium peroxide and ignited in an iron bomb: Three grms. of the sawdust, dried previously at 110° C., are mixed with 35 grms. of sodium peroxide, and the mixture is ignited by electrical means in a closed iron bomb about 4·5 inches in diameter and 5 inches deep. After the explosion the bomb is cooled, the contents are dissolved in water, the solution is transferred to a beaker, acidified with hydrochloric acid, and boiled for a few minutes. Two grms. of citric acid, 10 c.c. of concentrated nitric acid, and 5 c.c. of 8 per cent. ferric chloride solution are added, the solution is then rendered alkaline, an excess of 2 c.c. of concentrated ammonia is added, the whole is diluted to about 400 c.c., and titrated at 80° C. with standardised potassium ferrocyanide solution, using an external indicator consisting of equal parts of glacial acetic acid and glycerol. An excess of ammonia is necessary to keep the mixture from turning blue, but a large excess prolongs the end-point of the titration. The accuracy of the method was determined by adding a quantity of zinc chloride solution, equivalent to 0·0350 grm. of zinc, to 3 grms. of wood; six of these experiments were made. The quantity of zinc found varied from 0·0344 to 0·0351 grm. W. P. S.

Separation of Zirconium from Iron and Aluminium with the Aid of the Ammonium Salt of Nitrosophenylhydroxylamine ("Cupferron"). W. M. Thornton, jun., and E. M. Hayden, jun. (*Chem. News*, 1914, **110**, 153-155.)—Thornton has already (ANALYST, 1914, 332) shown how "cupferron" may be used to simplify the quantitative precipitation of titanium after iron has been separated from a tartaric acid solution. The principle of the method has now been extended to the separation of zirconium from iron and aluminium.

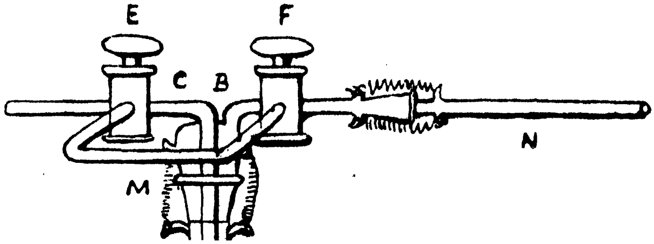
To the solution of the three metals tartaric acid is added in amount equal to five times the weight of the three bases to be held in solution. The mixture, which should not much exceed 100 c.c., is neutralised with ammonia and then acidified with 2 c.c. of dilute (1 : 1) sulphuric acid. Iron is reduced to the ferrous state by means of hydrogen sulphide, the solution rendered ammoniacal and hydrogen sulphide passed through it again until the precipitation of iron is complete, leaving the solution, however, alkaline to test-paper. The ferrous sulphide is filtered off and washed with very dilute, colourless ammonium sulphide, and the filtrate acidified with 50 c.c. dilute (1 : 1) sulphuric acid and boiled to expel hydrogen sulphide. To the thoroughly cooled solution, which should measure about 400 c.c., the "cupferron" reagent is added and the precipitate filtered off without delay, using mild suction. The filtrate is tested with more of the reagent, when an immediate white turbidity indicates unprecipitated zirconium. If sufficient "cupferron" was added in the first instance, addition of more to the filtrate gives rise only to a small white precipitate of nitrosophenylhydroxylamine, which disappears on standing, and is quite different in appearance from the zirconium compound. The latter is washed at least twenty times with dilute (1 : 10) hydrochloric acid, transferred with the filter to a platinum crucible, dried at 110° C., decomposed by cautious heating, and finally ignited for thirty minutes over a Meker burner. The extreme error of the method is 0·8 per cent. on the zirconium, when this metal is separated from three times its weight of iron and aluminium, or twenty times its weight of potassium.

In absence of iron, solutions of aluminium and zirconium have their acidity adjusted so that each 100 c.c. of the solution contains about 10 grms. of real sulphuric acid, and the zirconium is precipitated by means of "cupferron" as described. G. C. J.

APPARATUS, ETC.

Electro-Titrimetric Method and its Application to General Analytical Chemistry. F. H. H. van Suchtelen and A. Itano. (*J. Amer. Chem. Soc.*, 1914, **36**, 1793-1803.)—A brief summary is given of the results obtained in the application of this conductivity method (*cf.* ANALYST, 1909, **34**, 56; 1912, **37**, 282; 1913, **38**,

421) to the estimation of chlorine, silver, sulphuric acid, barium, uranium, phosphoric acid, potassium, calcium, and iron, as well as its use in acidimetric titrations. The end-points obtained were sharper than those exhibited by the usual indicators, and, when working with solutions of known strength, the results obtained agreed with those required by theory. The method was also applied to the estimation of chlorine and phosphoric acid in urine with satisfactory results. W. P. S.



Apparatus for the Combustion of very Volatile Liquids. E. Sernagiotto. (*Annali Chim. Applic.*, 1914, **1**, 195-198.)—The apparatus consists of a graduated tube, *A*, with a capacity of 3 to 5 c.c., into the neck of which is ground a stopper with tubes on the principle of Drechsel's washing-flask. The taps *E* and *F* have openings so that a current of air or gas may be passed through the tubes *C*, *B*, and *N*, by way of the side-tube *M*, without entering *A*. The detachable tube *N* is passed through the cork of the usual combustion-tube containing a copper spiral, etc. In practice a small quantity of the liquid is pipetted into *A*, the taps turned so as to open the communication through *M*, and the apparatus weighed. It is then connected with the combustion-tube by means of the tube *N*, whilst the other end is attached to a supply of oxygen. When the combustion-tube is hot, the taps *E*, *F* are turned so that a slow current of the gas passes through *A*. This evaporates and carries forward some of the liquid, and as soon as the quantity is shown by the graduations to be sufficient, the oxygen is replaced by a current of air for a few seconds, the taps then turned, and first oxygen and then air passed through the tubes *C*, *M*, *B*, *N*. Finally the apparatus is disconnected from *N*, and again weighed to obtain the amount of the liquid used in the combustion. In the case of hygroscopic liquids a drying agent (sodium sulphate or calcium chloride) is also placed in the tube *A*. C. A. M.

Apparatus for Analysis of Complex Gas Mixtures. G. B. Taylor. (*J. Ind. and Eng. Chem.*, 1914, **6**, 845-848.)—The apparatus described and illustrated embodies no novel principle, but combines several old ones in such a manner that the

United States Bureau of Mines approve of its recommendation not only for research, where great precision is required, but for many technical purposes where the slight loss of speed, compared with some other apparatus, may be more than compensated by the accuracy of the results obtainable. The number of liquid absorbents that can be used is unlimited, so that very complex mixtures can be analysed; but the most obvious use for the apparatus is for the analysis of gas mixtures containing higher paraffins than methane in addition to the latter, hydrogen, carbon monoxide, etc. Even commercial coal-gas may contain such quantities of higher paraffins that the disregard of this fact may lead to the hydrogen being under-estimated by 3 per cent. (on the gas), and the paraffins (assumed to be wholly methane) over-estimated by a like amount. The apparatus now described makes an accurate estimation of hydrogen possible, even in crude gas containing impurities which rapidly poison palladium catalysts. Similarly, the percentage and mean molecular weight of the paraffins may be accurately determined.

Use is made of the Bone and Wheeler method of absorption (ANALYST, 1908, **33**, 110), combined with the usual form of compensated burette, a copper oxide U-tube for the estimation of hydrogen and carbon monoxide, and a slow combustion pipette for burning hydrocarbons. The small quantity of reagent employed (3 c.c.) reduces to a minimum solution of gases not intended to be absorbed by a particular reagent. For example, 3 c.c. of fuming sulphuric acid has a scarcely measurable effect on the higher paraffins of natural gas or even of oil-gas, whereas the use of this reagent in a Hempel pipette to absorb olefines leads to large errors.

In analysing coal-gas, carbon dioxide is removed by potash, olefines by fuming sulphuric acid, and oxygen by alkaline pyrogallate in the usual manner. Hydrogen and carbon monoxide are oxidised by copper oxide at about 250° C., and separately estimated from the contraction due to combustion, and further contraction on treatment with potash. Finally, the paraffin hydrocarbons are estimated, and their mean molecular weight determined by mixing with oxygen, passing through an ordinary slow combustion pipette, and measurement of the contraction on combustion, and further contraction after treatment with potash. Pittsburg coal-gas, analysed in this way, is shown to contain 38·8 per cent. of hydrogen and 35·8 per cent. of paraffin (n in $C_nH_{2n+2} = 1·08$), whereas less accurate methods show 36 per cent. of hydrogen and 38·5 per cent. of "methane." G. C. J.

Instantaneous Thermostat and Smoke and Fume Monitors, Precipitators, and Recorders. W. W. Strong. (*J. Ind. and Eng. Chem.*, 1914, **6**, 848-849).—The lag of thermometers and of various forms of electrical pyrometers is well known. The instruments described in this paper are practically instantaneous in their operation, depending as they do on the intensity of ionisation of the gases in a furnace, which, in certain circumstances, is a measure of the temperature, in others, of the concentration of solid particles. When the gases from furnaces contain fumes, dust, or smoke particles, the ions tend to lose their charge to the particles, the conductivity of the furnace gases is greatly diminished in consequence, and this decrease in conductivity can be utilised to indicate the density of the fumes, smoke, or dust. This is the principle of the smoke and fume monitor described, which can be made to give an audible and visible indication when a certain concentration of dust is reached, for example, that corresponding to the limit tolerated in smoke stack discharge by the local by-laws. A recording type of instrument is also figured.

In the thermostat described, which depends on little or no dust or fumes being present, use is made of a piece of apparatus similar to the fume and smoke monitor, which contains two electrodes disposed within the gas. The thermostat is made to respond to variations in the conductivity of the gas in this gap—the conductivity being high where there is flame or high temperature, and low when there is little or no flame or low temperature. As there is no thermal conductivity or capacity involved, the apparatus is practically instantaneous. The relation between ionisation and temperature would have to be known before this instrument could be used to indicate absolute temperatures, but for many commercial purposes absolute temperatures are not required.

The fume precipitator consists of a transformer, taking 150 watts from any alternating lamp-socket, and rising up to 26,000 volts, together with a small electrode which serves to precipitate fumes, dust, and smoke by means of the corona discharge. The capacity of the instrument figured is about 100 cubic feet a minute, and fine smoke, such as cigarette smoke, is readily precipitated. Among other uses of the instrument, it is suggested that it would be useful for removing smoke or fumes from gases before leading these to a carbon dioxide recorder. G. C. J.

Thermo-Regulator for Thermostats at Temperatures very close to Room Temperature. C. W. R. Powell. (*J. Soc. Chem. Ind.*, 1914, **33**, 899-900.)

—The apparatus described has been devised for the purpose of maintaining a thermostat at a temperature differing by only a few degrees from that of the room in which it is situated; the heating is effected by one or more electric lamps, and, should the temperature rise too high, cooling is rapidly attained by running in ice water. A part of the regulator used (Fig. 1) is similar to the ordinary gas regulator. The bulb *L* is filled with toluene, and mercury is then poured in until it fills the lower part of *L* and the whole of the U-tube and capillary, *A*. The height of the mercury in the capillary is adjusted by the screw *H*. A glass-tube, *D*, carries ice-cold water into the chamber *I*, which is provided with two outlets, *B* and *T*. The switch for regulating the supply of heat is shown in Fig. 2. *R* and *S*, the two arms of a small electro-magnet, are joined to the terminals *R'* and *S'*. Two wires, *R''* and *S''* (Fig. 1), lead to *R'* and *S'*, the accumulator or cell being placed in the circuit. Heat is applied to the thermostat by suspending an electric globe-lamp in the bath; one wire from the lamp enters the switch at *C'* and leaves it at *D'*. *C'* is joined to the brass pin *C*, and *D'* to the fixed metal piece *D*. *N* is a small strip of wood pivoted at *P*; a strip of iron, *M*, is attached to *N* opposite the poles of the electro-magnet. A wire passes from *D* along *N*, and terminates in a brass pin opposite to *C*. The portion of this wire between *D* and the wooden bar is coiled, and acts as an extended spring to keep *N* away from the

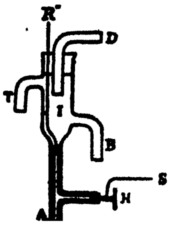


FIG. 1.

magnet when the latter is not magnetised. A rubber tube from the outlet *B* of the chamber *I* (Fig. 1) passes through *F* (Fig. 2), which is bored in the wooden base of the switch, and thence runs to the sink. The outlet *T* is in direct communication with the bath. When the temperature of the bath is correct, the level of the mercury in *A* (Fig. 1) stands just below the end of the wire *R''*; if the temperature rises, the mercury touches the end of the wire, thus completing the circuit, magnetising the electro-magnet *RS*, which then attracts the strip of iron *M* (Fig. 2), and breaks the lighting at *C*, thus interrupting the source of heat. At the same time the rubber tube leading from *B* (Fig. 1) is squeezed flat between the metal edge *E* and the small brass pillar *G*. The cold water, being now unable to escape to the sink through *B*, flows into the bath through *T*, and reduces the temperature accordingly. When the bath has been cooled to the proper temperature, the mercury thread falls below the end of the wire *R''*, the circuit to the magnet is broken, the spring *D* draws the wooden bar back to its original position, allowing the cold water to flow to the sink and again completing the circuit to the heating-lamp. The bath can be maintained within 0·5° C. of the desired temperature. W. P. S.

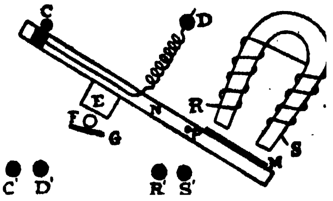


Fig. 2.

Rapid Laboratory Method of measuring Partial Vapour Pressures of Liquid Mixtures. M. R. Rosanoff, C. W. Bacon, and R. H. White. (*J. Amer. Chem. Soc.*, 1914, **36**, 1803-1825.)—In such work, what is really determined is the composition of the vapour which is in equilibrium with the liquid mixture. The method most often used consists in distilling off a small amount from the given mixture and analysing the distillate. As the distillate should be infinitely small for the results to be exact, and as this condition is unrealisable in practice, Rosanoff and others have devised a more exact method (*J. Amer. Chem. Soc.*, 1909, **31**, 448), which depends on passing a binary vapour of known constant composition through the liquid mixture until the b.-pt. and composition of the distillate become constant. The method is troublesome and is not recommended for ordinary use. It has served, however, to control experiments with a modification of the more usual method, now described and recommended.

Distilling apparatus is figured in the paper, which reduces reflux condensation to a minimum, the distilling vessel being surrounded by the escaping vapours passing through a jacket, which is in turn surrounded by a water-bath, maintained a few degrees above the maximum b.-pt. of the liquid. The liquid is heated by an electrical heater. The principle of the method is the collection of a number of fractions, their analysis by refractometric measurements or other means, and the plotting of the results on a system of rectangular co-ordinates. By slight extrapolation, the composition of the first, infinitely small, drop of distillate may be estimated with satisfactory accuracy. The same data serve to construct a second curve, showing the composition of the residue in the still when any definite percentage has been distilled, and the two curves together give the composition of the vapour which is in equilibrium with liquid mixtures of widely varying composition. G. C. J.

REVIEWS.

HISTORY OF THE INSTITUTE OF CHEMISTRY, 1877-1914. By RICHARD B. PILCHER, Registrar and Secretary. Price 5s. net.

ON the eve of the departure of the Institute of Chemistry from its old quarters to a new and permanent home in Russell Square, the Council has inspired or encouraged in its Registrar the idea of compiling a detailed history of the Institute and its work from its foundation to the present time, and Mr. Pilcher has thrown himself into the task with the thoroughness which we have learned to expect in the case of any matter which he takes in hand. The result is a volume of 300 pages containing many reminiscences which will afford interesting reading for those of the Fellows whose recollections go back to our early struggles to organise ourselves as a recognised profession. At the same time it affords the younger generation of Fellows an opportunity of learning something of the arduousness of the work done by those who acted as pioneers.

The progress and continuous development of that work, of which even the later stages are perhaps less widely and fully realised than they should be, are set forth in the form of a fairly complete and connected narrative. It is true that a large part of this, especially of the later portion of the history, has been already recorded in the Proceedings, which are regularly issued in pamphlet form; but pamphlet literature often remains unassimilated by those to whom it is addressed. It may be delivered on a busy day, and put under the fatal paperweight for later perusal, and then prematurely docketted away as read; or it may meet the easy fate of more ephemeral publications. That this sometimes happens to the Proceedings of the Institute seems almost a necessary inference from the fact that one still occasionally —though happily with increasing rarity—comes across Fellows who ask, with an air of honest inquiry, "What does the Institute do in return for the tax that it annually levies on us?" In all ranks there are, of course, some chronic and hopeless malcontents, but any unconvinced yet open-minded framer of such a question who will read Mr. Pilcher's history ought not to find himself in disagreement with the claims set forth in the paragraphs with which he brings it to a close. These paragraphs run as follows:

"It has been shown that, at the time of the foundation of the Institute, very few universities or colleges in this country provided satisfactory preparation for the profession of chemistry, and it may be claimed that the Institute, by its examinations, in which a very high standard has been maintained, has taken no small part in the development of systematic chemical education. It has succeeded in forming a register of competent reliable chemists for the service of the community in the various branches of work—as consultants and analysts in private practice, in industry, and in Government and municipal laboratories, and as professors and teachers of chemistry.

"In connection with legislation, the Institute has endeavoured to assist the Government in all matters on which the science of chemistry has a bearing, whilst the Government in return has in a large measure recognised the work and qualifications of the Institute, though, as has been indicated, the importance of the services of the chemist is not yet as fully realised as the Institute would deem desirable.

"By the Conferences on professional matters and by the work of the Censors, the Institute has fostered among the members a sense of mutual responsibility in their relations to the public and to one another as professional men. By the Lectures Scheme, students and members are brought into touch with men of experience, and thus obtain an insight into the actual conditions of practice. Through the Appointments Register, the Institute has provided a means whereby authorities and employers can secure competent chemists, and newly qualified Associates and the younger members generally can be informed of suitable vacancies. In connection with this department of its work, the Institute has the assistance of the honorary corresponding secretaries, who are able to advise Fellows and Associates with regard to appointments in India and the overseas Dominions. The library, further, provides a steadily growing collection of standard works of reference, which has proved of much service to the Fellows and Associates, to students, and to candidates for the various examinations.

"The Council and officers of the Institute have been ever ready to consider suggestions for the furtherance of its work, and, so far as they have been able, have extended the privileges of the Fellows, Associates, and students. The office, too, has become increasingly useful to members and others requiring information and advice on matters of professional interest.

"It is difficult to realise what the position of professional chemists would have been at the present day if no such institution had been founded, and no attempt been made to bring together in one body those who practise in a calling now so necessary to the affairs of everyday life and the progress of civilisation. It is clearly to the interest of all such chemists that they should co-operate in furthering the welfare of their profession, and that there should exist a representative body to which the Government, the community, and the members themselves should be able to turn for advice and assistance. It is hoped, therefore, that the Institute will become more and more representative of the whole profession.

"With the loyal co-operation of the Fellows and Associates, the Council and officers look forward with full confidence to its continued and steady advance both in prestige and influence."

Had these paragraphs been printed as a thesis at the beginning of the volume, most readers would say that the thesis had been well maintained.

It was in 1875 that the first meeting of chemists was convened to discuss the advisability of founding an institution to promote the education and protect the interests of the chemical profession, and for nearly two years heated controversy went on as to the best mode of effecting the organisation the desirability of which was admitted on all sides. Many considered that the Chemical Society should, if possible, assume some such functions as those which were ultimately allotted to the Institute, and it was also suggested that the Royal School of Mines might expand its scope in some such direction. For a time there was a strong feeling among many practising analysts that the best body to form the nucleus of the proposed association or institution would be the Society of Public Analysts, which was then already in existence and in vigorous work. When ultimately the Institute of Chemistry took shape, there were still among the members of the Society of Public Analysts some who viewed it jealously. But most of the members gave it their cordial co-operation, and most, if not all, of those who had been prominently opposed to its original constitution, finally enrolled themselves as members of the new body. The history of much of the controversy will be found in the pages of Mr. Pilcher's book.

One of the old episodes in the history of the Institute which should be most interesting to members of the Society of Public Analysts is connected with the report of a special committee of the Council of the Institute which Mr. Pilcher has abstracted from the minute-books for 1889, when the Institute was twelve years old. Some of the matter in this report is now made public for the first time. The Committee was appointed to consider the question of the holding of periodical conferences in order to bring the members of the Institute into frequent intercourse and to advance professional interests in a more practical and efficient manner than was possible while the Institute restricted its activity, as it then did, solely to the exercise of examinational and diploma-granting functions. Among the subjects suggested by the Committee for discussion were the training of students, the status and conduct of the professional chemist and his relations towards individual clients and public authorities or corporations, and the actual work of the analytical and consulting chemist, such as the description and consideration of new methods of analysis and the inferences to be drawn from analytical data. Abstracts of the report will be found on pages 97 and 98. It will be seen that the report pointed out that some of the objects for which the Society of Public Analysts had been founded had been by that time to a large extent realised, and suggested that the time had come when its work might be regarded as falling within the legitimate scope of the functions of the Institute, and that general and material advantage both to Public Analysts and to the profession at large might ensue if the smaller society could be induced to merge itself in the larger. There was a suggestion that under such conditions a Committee on State Chemistry, formed of members of the Council of the Institute, could specially watch over matters affecting the relationship of the professional chemist to Government Departments and public authorities, and over matters which might be the subject of chemical legislation; and also that a journal might be published by the Institute, very much on the lines of the ANALYST as it exists to-day. Under the proposed amalgamation, meetings such as those then held by the Society of Public Analysts, but with wider aims and objects, were to be held regularly under the auspices of the Institute; and the Committee finally recommended "that the Council should request the President of the Institute to confer with the President of the Society of Public Analysts, with the view of ascertaining whether such a fusion of the aims and interests of both bodies was likely to commend itself to the members as practicable, and as of mutual advantage." It is scarcely necessary to say that, while some of the suggestions in the report have since borne good fruit in the Institute, the main proposal fell through.

Here the record ends, being probably as complete as it could be made from the minute-books. At this distance of time, however, no harm can be done by giving a further instalment of narrative relating to a subject which might have been very momentous in the history of both the bodies concerned. The writer of this revi was a member of the Conference Committee responsible for the report, much which was indeed drafted by his own hand with the concurrence of the th President of the Society of Public Analysts, Dr. Adams, and of a few other leadin members of the Society, of which he and Mr. Hehner were then the Secretaries Only seven others survive out of the twenty-four officers and members of Counci of the Society of Public Analysts of that year.

When the report was presented to the Council of the Institute, a strong antagonism to its main proposal was immediately evinced by some of its members who were out of touch and sympathy with the Society of Public Analysts, and wh read into the report a veiled attempt on the part of the Society to "capture" an "run" the Institute for its own narrower aims. On the other hand, perhaps owin to the subject having been handled with an excess of discretion, there becam manifest an invincible suspicion on the part of some prominent members of the Society of Public Analysts that the recommendation of amalgamation was the resul of a Machiavellian plot on the part of the President of the Institute, Dr. Bell (afterwards Sir James Bell), to annihilate the Society of Public Analysts, which notoriously had been long on anything but good terms with the Inland Revenue Laboratory, of which Dr. Bell was the head. That there had been strong antagonism between Public Analysts as a body and the Inland Revenue chemists is a matter of open history. It was strongly held among the Public Analysts that there was not a little grave incompetency in the Inland Revenue Laboratory for the duties which the Legislature had thrown upon it as the Reference Laboratory under the Sale of Food and Drugs Act, and it is perhaps sufficient now to say that these views, if sometimes exaggerated, were not groundless. At the same time, the Public Analysts themselves, who, unaided, were called upon to explore and develop the chemistry of food analysis, were not immune from occasional errors, the outcome of imperfect experience. The chemists of the Inland Revenue Laboratory and the Public Analysts should from the first have worked together, sympathetically, as they happily do to-day; but did not. The present writer's recollection takes him through all the stages of th sad warfare of those early days, very many of the leading combatants in which have nce passed away. Probably the few who are left would for the most part agree i the retrospective view that, while most of the trouble may have been due to the attitude of the Government Laboratory and to its then non-receptivity of new ideas, and to a perhaps mistaken construction of its functions, there was at the same time not a little lack of tact, and sometimes of taste, on the part of some of the Public Analysts, in the mode of presentment of the grievances under which they suffered. Dr. Bell, personally, cherished ideas of co-operation with the Public Analysts, although these views, unfortunately, appeared not to be fully shared by some of the senior members of his staff; and there are, no doubt, even now some of the then members of the Society of Public Analysts still among us who will be interested in learning for the first time that the framing of the report of the Institute Committee was really seized as a happy opportunity for mooting a proposal which had been previously informally discussed in private conference between Dr. Bell and some of the Public Analysts, as a means of drawing together the Government Laboratory the Public Analysts on the neutral ground of the Institute, and with a view to ring the breach which then seemed otherwise difficult to mend. It was an st attempt on both sides to achieve a certainly desirable end, and Dr. Bell's tion as President of the Institute seemed to afford a golden opportunity of ending troubles. But when the scheme was finally discussed, the Council of the Institute—the writer's memory serves him well—was almost equally divided, and it was clear at the adoption of the chief recommendation of the report would have led to resignations from the Institute of not a few Fellows, including both members and non-members the Society of Public Analysts. Under these circumstances Dr. Bell was wise enough counsel the withdrawal of the recommendation. Some of us at the time were sappointed, but there can now be scarcely any of the members of the Society of ublic Analysts who, having regard to the Society's great development during the st five-and-twenty years, would be otherwise than glad that the proposed amalgaation fell through. There may since have been, here and there, a little overlapping the work of the Institute and of the Society, but, on the whole, their main work is distinct, and neither would probably have been able to develop the full scope of utility now attained by both had not the essential functions which they respectively perform been kept apart. As regards matters which properly fall within the purview of both bodies, their Councils have for a long time past worked in co-operation, with the aid of jointly appointed committees, to the mutual advantage of the members of both bodies and the greater good of the profession. BERNARD DYER.

THE SIMPLER NATURAL BASES. By GEORGE BARGER. Longmans, Green and Co. 1914. Price 6s. net.

This little book, one of the latest additions to the series of Monographs on Biochemistry, edited by Dr. Plimmer and Professor Hopkins, fills a distinct void in chemical literature, and Dr. Barger's name on the title-page is a sufficient guarantee that the work has been well done.

One can well believe that at the outset the author must have found the choice of a title no easy task. The great diversity and heterogeneous character of the organic bases, and the fact that they do not constitute a well-defined chemical group, must clearly have cast upon the author the necessity of devising some guiding principle, and the responsibility of deciding whether certain basic substances should, or should not, be included within the covers of his work. In the writer's opinion Dr. Barger's selection of subject-matter, although necessarily of an arbitrary character, has been judicious, and well calculated to insure the usefulness of the book to those for whom it has been primarily written—the workers in the borderlands of chemistry and biology.

For the purposes of his book, the author defines a base as a substance which is precipitated by phospho-tungstic acid, and those who have had much practical experience in this department of chemistry will be inclined to agree that many a worse definition might have been put forward. Add to this the limitation of the term to those substances of comparatively simple constitution and of wide occurrence which are in close genetic relationship to the proteins and phosphatides, and the scope of the book will be apparent.

The first three chapters deal with the amines derived from the breaking down of proteins, the amino-acids, and the betaines, chapter iv. with choline and some allied substances, and chapter v. with creatine, creatinine, glycocyamine, and guanidines. Owing to its very powerful physiological activity and its extensive application, the author has included adrenaline, and has devoted a good deal of space to this interesting substance. In addition to these a few bases of unknown constitution, such as spermine, vitamine, and sepsine, are briefly dealt with.

Whilst the book may be expected to appeal chiefly to the biochemist, it contains much matter of great and growing importance to analysts, and particularly to those who are concerned with the examination of natural and artificial food products. To such, the chapter (in the form of a lengthy appendix) on the Practical Chemical Methods employed for the Separation, Isolation, and Identification of Individual Bases, cannot fail to prove exceedingly helpful. Selected methods which have stood the test of experience are given, and this section is, in the writer's opinion, one of the best in the book.

An exhaustive bibliography and a good index add greatly to the value of this little work. A. CHASTON CHAPMAN.

FUEL, GASEOUS, SOLID, AND LIQUID. By J. H. COSTE, F.I.C., and E. R. ANDREWS, F.I.C. London: Chas. Griffin and Co. Price 6s. net.

This book forms a companion volume to that previously published by Mr. Coste on the Calorific Power of Gas, and is divided into two parts, the first dealing with the sampling, chemical, and physical analysis of gaseous, liquid, and solid fuel, and the second with their calorimetry.

Within the limits imposed by the size of the book, a good description of the methods of sampling and analysis is given in the first part, and this will prove of use to those engaged in these analyses. Opinions will of course differ as to the relative amount of space which should be given to the different parts of the subject, and to the reviewer it appears that it would have been more advantageous to compress somewhat the rather extended consideration given to the determination of moisture, and to increase that allowed for the discussion of the other analytical methods, especially in the case of liquid fuel; and in the case of solid fuel to a more complete account of the determination of nitrogen in coal, which is of such importance, when the fuel is gasified in ammonia-recovery producers.

The treatment of calorimetry and of the various calorimeters and the practical description of the actual carrying out of the calorimetric tests with solids and liquids is well done, showing a detailed acquaintance with the difficulties which may be met with and the pitfalls which have to be avoided. The portion relating to gas calorimetry is very much condensed, this portion of the subject being dealt with at length by Mr. Coste in the previous book on this subject.

While the book will be very useful to any chemist engaged in making calorific determinations, its value would be considerably enhanced if there were added some discussion of the value of the determination of the calorific power of fuels from a chemical-engineering point of view. Fundamental as this value is, it is by no means the only point to be considered in practice, and there is considerable danger of over-estimating its importance, unless the other factors receive proper consideration. The authors do incidentally make a number of statements which show that this consideration is not absent from their minds, but a fuller consideration of the matter is advisable; although the book is primarily intended for trained chemists, it will be consulted by engineers, and it is also most important that the chemist who is carrying out these determinations, and who may have to make reports recommending the adoption or rejection of a fuel, should have as full an understanding as possible of these other factors.

The book is commendably free from misstatements and printer's errors. The following may be mentioned as requiring amendment in a new edition: On p. 15, referring to the gaseous constituents of spent gases, only CO and CH_4 are mentioned as possible combustible constituents, whereas hydrogen may be, and is, often present. It is also stated that CS_2 vapour is removed from a gaseous mixture by caustic potash, whereas this is only the case, except to a very limited extent, when *alcoholic* potash is employed. On pp. 124 and 125, the date of Fahrenheit's introduction of his thermometric scale is given as 1820, instead of 1714; and on p. 137 it is stated that the kilogramme-calorie = 1·000 gramme or small calories, instead of 1000 gramme calories. In a future edition a much more extended index would add to the usefulness of the book, the present index being very incomplete. H. G. Colman